Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*Z*)-2-(2-Aminothiazol-1-ium-4-yl)-2-(*tert*-butoxycarbonylmethoxyimino)-acetate monohydrate

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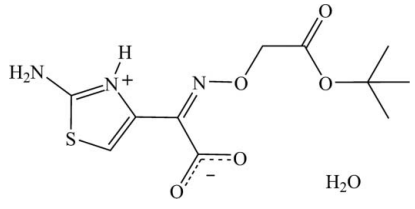
Received 23 July 2007; accepted 5 August 2007

Key indicators: single-crystal X-ray study; T = 223 K; mean σ(C–C) = 0.002 Å; R factor = 0.031; wR factor = 0.084; data-to-parameter ratio = 17.1.

In the title compound, $C_{11}H_{15}N_3O_5S\cdot H_2O$, the amino group is coplanar with the thiazole ring. The dihedral angle between the thiazole ring and the adjacent carboxylate group is 74.19 (4)°. O—H···O and N—H···O hydrogen bonds link the molecules into two-dimensional networks parallel to the (100) plane, and π–π interactions exist between the thiazole rings of inversion-related molecules, with a centroid–centroid separation of 3.5965 (6) Å.

Related literature

For synthesis details, see: Furlenmeier *et al.* (1987). For general background, see: Quintiliani (1996); Lynch *et al.* (1999); Toplak *et al.* (2003). For related structures, see: Laurent *et al.* (1981); Yoshida *et al.* (1989). For related literature, see: Allen (2002).



Experimental

Crystal data

$C_{11}H_{15}N_3O_5S\cdot H_2O$
M_r = 319.34
Monoclinic, $P2_1/c$
a = 10.4504 (2) Å
b = 11.2896 (3) Å
c = 13.0965 (3) Å
β = 100.407 (1)°
V = 1519.72 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 223 (2) K
0.70 × 0.70 × 0.57 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.848, T_{max} = 0.874
14426 measured reflections
3446 independent reflections
3253 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.084
S = 1.04
3446 reflections
202 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O4^{i}	0.87	2.28	2.8647 (15)	125
N1—H1A···O1^{i}	0.87	2.44	3.0683 (15)	130
N1—H1B···O1W^{ii}	0.87	1.90	2.7443 (16)	162
N3—H12···O1^{i}	0.88 (2)	1.89 (2)	2.6914 (13)	151 (2)
O1W—H1W···O2^{iii}	0.89 (3)	1.79 (3)	2.6637 (16)	166 (2)
O1W—H2W···O5	0.81 (3)	2.20 (3)	2.9840 (16)	162 (2)

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

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: *SHELXTL*.

The author acknowledges financial support from Zhejiang Police College, China.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BI2222).

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Bruker (2002). *SADABS*, *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Furlenmeier, A., Hofheinz, W. & Hubschw, E. (1987). US Patent 4 652 651.
Laurent, G., Parmentier, C., Evrard, G. & Durant, F. (1981). *Acta Cryst.* B**37**, 974–976.
Lynch, D. E., Nicholls, L. J., Smith, G., Byriel, K. A. & Kennard, C. H. L. (1999). *Acta Cryst.* B**55**, 758–766.
Quintiliani, R. (1996). *Curr. Ther. Res. Clin. Exp.* **57**, 892–912.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (2003). *SADABS*. Version 2.10. University of Göttingen, Germany.
Toplak, R., Lah, N., Volmajer, J., Leban, I. & Majcen Le Maréchal, A. (2003). *Acta Cryst.* C**59**, o502–o505.
Yoshida, A., Moroi, R., Arimoto, M. & Furukawa, M. (1989). *Anal. Sci.* **5**, 785–786.

  doi:10.1107/S1600536807038469

supplementary materials

Acta Cryst. (2007). E**63**, o3728 [**doi:10.1107/S1600536807038469**]

(*Z*)-(2-Aminothiazol-1-ium-4-yl)-2-(*tert*-butoxycarbonylmethoxyimino)acetate monohydrate

X.-W. Cheng

Comment

2-Aminothiazole compounds have been extensively studied because of their biological and industrial applications (Lynch *et al.*, 1999; Toplak *et al.*, 2003). A search of the Cambridge Structural Database (CSD, Version 5.28, May 2007; Allen, 2002) reveals that there are 127 crystal structures containing the 2-aminothiazole moiety. The title compound is an important intermediate of cefixime, which was the first oral third-generation cephalosporin available in Japan, The United States, Europe and other regions (Quintiliani, 1996). Crystal structures of some cephalosporin intermediates which contain a 2-aminothiazole group have been reported (Yoshida *et al.*, 1989; Laurent *et al.*, 1981).

In the title compound, the thiazole ring is planar to within 0.0047 (7) Å. The amino group and atom C4 are coplanar with the thiazole ring, with atoms N1 and C4 deviating from the thiazole plane by −0.0039 (3) and −0.075 (2)\ %A, respectively. The dihedral angle between the thiazole ring and the adjacent carboxylate group is 74.19 (4) °. The C3/C4/N2/O3 plane is twisted away from the thiazole plane by 3.20 (5) °, while the dihedral angle between the C3/C4/N2/O3 plane and adjacent carboxylate group is 77.39 (4) °.

The crystal structure is stabilized by intermolecular O—H···O and N—H···O hydrogen bonds (Table 1) involving the O atoms of the water molecules, carboxylate groups and ester groups. These hydrogen bonds link the molecules into two-dimensional networks parallel to the (100) planes (Fig. 2). The thiazole rings of the inversion-related molecules at (x, y, z) and ($2 - x$, $-y$, $1 - z$) are stacked with their centroids separated by a distance of 3.5965 (6) Å, indicating π-π interactions.

Experimental

The title compound was prepared according to the literature method (Furlenmeier *et al.*, 1987). Crystals suitable for X-ray analysis were obtained by slow evaporation of an 2-propanol solution at 223 K

Refinement

H atoms bound to N or O atoms were located from a difference Fourier map and refined freely. H atoms bound to C atoms were positioned geometrically (C—H = 0.94–0.98 Å) and refined using a riding model, with U_{iso}(H) = 1.2 or 1.5U_{eq}(C).

Figures

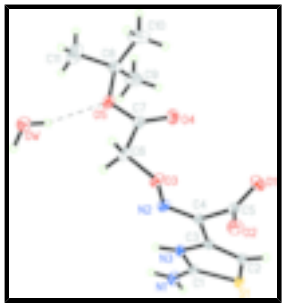


Fig. 1. Molecular structure of the title compound, showing 30% probability displacement for non-H atoms. The dashed line denotes an O—H···O hydrogen bond.

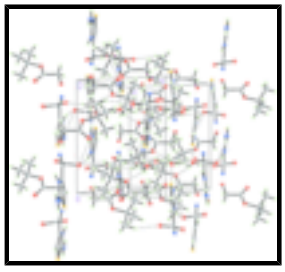


Fig. 2. Packing diagram. Dashed lines indicate intermolecular hydrogen bonds.

(*Z*)-(2-Aminothiazol-1-ium-4-yl)-2-(*tert*-butoxycarbonylmethoxyimino)acetate monohydrate

Crystal data

$C_{11}H_{15}N_3O_5S·H_2O$	F_{000} = 672
M_r = 319.34	D_x = 1.396 Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 459-461 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 10.4504 (2) Å	Cell parameters from 3446 reflections
b = 11.2896 (3) Å	θ = 2.0–27.5º
c = 13.0965 (3) Å	μ = 0.24 mm^{-1}
β = 100.407 (1)º	T = 223 (2) K
V = 1519.72 (6) Å^3	Block, colourless
Z = 4	0.70 × 0.70 × 0.57 mm

Data collection

Bruker SMART CCD diffractometer	3446 independent reflections
Radiation source: fine-focus sealed tube	3253 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.021
T = 223(2) K	θ_{max} = 27.5º
φ and ω scans	θ_{min} = 2.0º
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	h = −13→13
T_{min} = 0.848, T_{max} = 0.874	k = −14→14
14426 measured reflections	l = −17→17

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.031$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.084$	$w = 1/[\sigma^2(F_o^2) + (0.0414P)^2 + 0.4932P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} = 0.001$
3446 reflections	$\Delta\rho_{max} = 0.35$ e Å^{-3}
202 parameters	$\Delta\rho_{min} = -0.19$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2$sigma(F^2) is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N2	0.88830 (10)	0.35059 (8)	0.40598 (8)	0.0270 (2)
N1	0.76747 (12)	−0.00740 (10)	0.59144 (9)	0.0351 (2)
H1A	0.7563	0.0458	0.6371	0.042*
H1B	0.7510	−0.0815	0.6020	0.042*
C3	0.87952 (10)	0.14516 (10)	0.38807 (8)	0.0232 (2)
C4	0.90625 (10)	0.26202 (10)	0.34927 (9)	0.0233 (2)
C1	0.80927 (11)	0.02414 (10)	0.50659 (9)	0.0257 (2)
C2	0.88474 (11)	0.04012 (10)	0.34035 (9)	0.0259 (2)
H2	0.9114	0.0305	0.2760	0.031*
C5	0.94464 (12)	0.27073 (9)	0.24220 (9)	0.0259 (2)
C6	0.85040 (12)	0.55092 (11)	0.40047 (11)	0.0323 (3)
H6A	0.8892	0.6273	0.3879	0.039*
H6B	0.8586	0.5406	0.4757	0.039*
C7	0.70753 (12)	0.55079 (10)	0.35059 (10)	0.0296 (2)
C8	0.50370 (13)	0.66070 (13)	0.35446 (12)	0.0413 (3)
C9	0.41658 (16)	0.55500 (17)	0.35991 (16)	0.0568 (4)
H9A	0.4360	0.4940	0.3128	0.085*

H9B	0.3263	0.5789	0.3402	0.085*
H9C	0.4315	0.5242	0.4302	0.085*
C10	0.4928 (2)	0.7057 (2)	0.24477 (18)	0.0706 (6)
H10A	0.5100	0.6414	0.2000	0.106*
H10B	0.5557	0.7685	0.2431	0.106*
H10C	0.4058	0.7361	0.2207	0.106*
C11	0.47690 (19)	0.7585 (2)	0.4269 (2)	0.0842 (8)
H11A	0.4844	0.7273	0.4967	0.126*
H11B	0.3897	0.7889	0.4038	0.126*
H11C	0.5394	0.8219	0.4265	0.126*
O5	0.64267 (9)	0.62911 (8)	0.39814 (8)	0.0370 (2)
O3	0.91826 (9)	0.45830 (7)	0.35939 (7)	0.0317 (2)
O2	1.06094 (9)	0.28914 (9)	0.23883 (7)	0.0359 (2)
N3	0.83732 (9)	0.13513 (8)	0.48304 (7)	0.0244 (2)
H12	0.8441 (17)	0.1918 (17)	0.5299 (15)	0.047 (5)*
O1	0.85201 (10)	0.25782 (9)	0.16842 (7)	0.0384 (2)
O4	0.66198 (10)	0.48971 (9)	0.27812 (8)	0.0405 (2)
S1	0.83619 (3)	−0.07545 (2)	0.41203 (2)	0.02844 (9)
O1W	0.75233 (14)	0.75009 (10)	0.59734 (11)	0.0578 (3)
H1W	0.814 (2)	0.724 (2)	0.649 (2)	0.079 (7)*
H2W	0.726 (2)	0.704 (2)	0.551 (2)	0.075 (7)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N2	0.0313 (5)	0.0227 (5)	0.0287 (5)	0.0010 (4)	0.0097 (4)	0.0022 (4)
O1W	0.0694 (8)	0.0357 (6)	0.0586 (8)	0.0060 (5)	−0.0141 (6)	−0.0073 (5)
N1	0.0523 (6)	0.0284 (5)	0.0298 (5)	−0.0008 (5)	0.0208 (5)	0.0018 (4)
C3	0.0252 (5)	0.0252 (5)	0.0196 (5)	0.0024 (4)	0.0056 (4)	0.0022 (4)
C4	0.0245 (5)	0.0237 (5)	0.0220 (5)	0.0019 (4)	0.0052 (4)	0.0023 (4)
C1	0.0292 (5)	0.0256 (5)	0.0238 (5)	0.0021 (4)	0.0082 (4)	0.0009 (4)
C2	0.0335 (5)	0.0253 (5)	0.0204 (5)	0.0014 (4)	0.0090 (4)	0.0017 (4)
C5	0.0365 (6)	0.0191 (5)	0.0237 (5)	0.0025 (4)	0.0100 (4)	0.0036 (4)
C6	0.0349 (6)	0.0226 (5)	0.0409 (7)	0.0027 (4)	0.0111 (5)	−0.0054 (5)
C7	0.0368 (6)	0.0250 (5)	0.0292 (6)	0.0026 (4)	0.0117 (5)	0.0010 (4)
C8	0.0324 (6)	0.0432 (7)	0.0462 (8)	0.0099 (5)	0.0015 (6)	−0.0041 (6)
C9	0.0383 (8)	0.0649 (11)	0.0648 (11)	−0.0013 (7)	0.0029 (7)	0.0111 (9)
C10	0.0623 (11)	0.0748 (13)	0.0739 (13)	0.0211 (10)	0.0102 (9)	0.0350 (11)
C11	0.0440 (9)	0.0921 (16)	0.1097 (18)	0.0300 (10)	−0.0048 (10)	−0.0523 (14)
O5	0.0321 (4)	0.0375 (5)	0.0404 (5)	0.0081 (4)	0.0041 (4)	−0.0112 (4)
O3	0.0376 (4)	0.0201 (4)	0.0412 (5)	0.0025 (3)	0.0176 (4)	0.0016 (3)
O2	0.0363 (5)	0.0404 (5)	0.0350 (5)	0.0012 (4)	0.0166 (4)	0.0057 (4)
N3	0.0307 (5)	0.0231 (4)	0.0209 (5)	0.0015 (4)	0.0088 (4)	0.0002 (3)
O1	0.0459 (5)	0.0456 (5)	0.0228 (4)	−0.0057 (4)	0.0039 (4)	0.0057 (4)
O4	0.0448 (5)	0.0437 (5)	0.0334 (5)	0.0024 (4)	0.0075 (4)	−0.0098 (4)
S1	0.04030 (17)	0.02140 (14)	0.02611 (15)	−0.00018 (10)	0.01262 (12)	−0.00021 (10)

Geometric parameters (Å, °)

N2—C4	1.2795 (15)	C6—H6A	0.980
N2—O3	1.4206 (12)	C6—H6B	0.980
O1W—H1W	0.89 (3)	C7—O4	1.1998 (16)
O1W—H2W	0.81 (3)	C7—O5	1.3347 (15)
N1—C1	1.3145 (15)	C8—O5	1.5046 (15)
N1—H1A	0.870	C8—C10	1.508 (3)
N1—H1B	0.870	C8—C9	1.510 (2)
C3—C2	1.3461 (16)	C8—C11	1.514 (2)
C3—N3	1.3973 (14)	C9—H9A	0.970
C3—C4	1.4589 (15)	C9—H9B	0.970
C4—C5	1.5297 (15)	C9—H9C	0.970
C1—N3	1.3355 (15)	C10—H10A	0.970
C1—S1	1.7328 (12)	C10—H10B	0.970
C2—S1	1.7358 (11)	C10—H10C	0.970
C2—H2	0.940	C11—H11A	0.970
C5—O2	1.2417 (15)	C11—H11B	0.970
C5—O1	1.2466 (15)	C11—H11C	0.970
C6—O3	1.4225 (14)	N3—H12	0.88 (2)
C6—C7	1.5177 (17)		
C4—N2—O3	110.66 (9)	O5—C8—C9	110.08 (12)
H1W—O1W—H2W	117 (2)	C10—C8—C9	111.63 (15)
C1—N1—H1A	120.0	O5—C8—C11	102.11 (12)
C1—N1—H1B	120.0	C10—C8—C11	111.13 (18)
H1A—N1—H1B	120.0	C9—C8—C11	111.41 (16)
C2—C3—N3	112.81 (10)	C8—C9—H9A	109.5
C2—C3—C4	127.48 (10)	C8—C9—H9B	109.5
N3—C3—C4	119.64 (10)	H9A—C9—H9B	109.5
N2—C4—C3	116.56 (10)	C8—C9—H9C	109.5
N2—C4—C5	124.90 (10)	H9A—C9—H9C	109.5
C3—C4—C5	118.42 (9)	H9B—C9—H9C	109.5
N1—C1—N3	124.73 (11)	C8—C10—H10A	109.5
N1—C1—S1	123.37 (9)	C8—C10—H10B	109.5
N3—C1—S1	111.90 (8)	H10A—C10—H10B	109.5
C3—C2—S1	111.76 (8)	C8—C10—H10C	109.5
C3—C2—H2	124.1	H10A—C10—H10C	109.5
S1—C2—H2	124.1	H10B—C10—H10C	109.5
O2—C5—O1	128.32 (11)	C8—C11—H11A	109.5
O2—C5—C4	117.63 (10)	C8—C11—H11B	109.5
O1—C5—C4	114.05 (10)	H11A—C11—H11B	109.5
O3—C6—C7	110.82 (10)	C8—C11—H11C	109.5
O3—C6—H6A	109.5	H11A—C11—H11C	109.5
C7—C6—H6A	109.5	H11B—C11—H11C	109.5
O3—C6—H6B	109.5	C7—O5—C8	121.08 (10)
C7—C6—H6B	109.5	N2—O3—C6	107.65 (9)
H6A—C6—H6B	108.1	C1—N3—C3	113.53 (9)
O4—C7—O5	125.98 (12)	C1—N3—H12	121.0 (12)

O4—C7—C6	124.05 (11)	C3—N3—H12	124.2 (12)
O5—C7—C6	109.96 (10)	C1—S1—C2	89.99 (5)
O5—C8—C10	110.08 (13)		
O3—N2—C4—C3	179.80 (9)	O4—C7—O5—C8	−8.4 (2)
O3—N2—C4—C5	−4.32 (15)	C6—C7—O5—C8	170.59 (11)
C2—C3—C4—N2	176.36 (11)	C10—C8—O5—C7	−56.72 (18)
N3—C3—C4—N2	−0.33 (15)	C9—C8—O5—C7	66.76 (17)
C2—C3—C4—C5	0.20 (17)	C11—C8—O5—C7	−174.82 (16)
N3—C3—C4—C5	−176.49 (9)	C4—N2—O3—C6	157.27 (10)
N3—C3—C2—S1	0.33 (13)	C7—C6—O3—N2	−77.04 (12)
C4—C3—C2—S1	−176.55 (9)	N1—C1—N3—C3	−179.79 (11)
N2—C4—C5—O2	79.82 (15)	S1—C1—N3—C3	0.87 (12)
C3—C4—C5—O2	−104.37 (12)	C2—C3—N3—C1	−0.79 (14)
N2—C4—C5—O1	−100.26 (13)	C4—C3—N3—C1	176.36 (10)
C3—C4—C5—O1	75.55 (13)	N1—C1—S1—C2	−179.92 (11)
O3—C6—C7—O4	−8.02 (18)	N3—C1—S1—C2	−0.57 (9)
O3—C6—C7—O5	172.96 (10)	C3—C2—S1—C1	0.12 (9)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···O4^{i}	0.87	2.28	2.8647 (15)	125
N1—H1A···O1^{i}	0.87	2.44	3.0683 (15)	130
N1—H1B···O1W^{ii}	0.87	1.90	2.7443 (16)	162
N3—H12···O1^{i}	0.88 (2)	1.89 (2)	2.6914 (13)	151 (2)
O1W—H1W···O2^{iii}	0.89 (3)	1.79 (3)	2.6637 (16)	166 (2)
O1W—H2W···O5	0.81 (3)	2.20 (3)	2.9840 (16)	162 (2)

Symmetry codes: (i) *x*, −*y*+1/2, *z*+1/2; (ii) *x*, *y*−1, *z*; (iii) −*x*+2, −*y*+1, −*z*+1.

Fig. 1

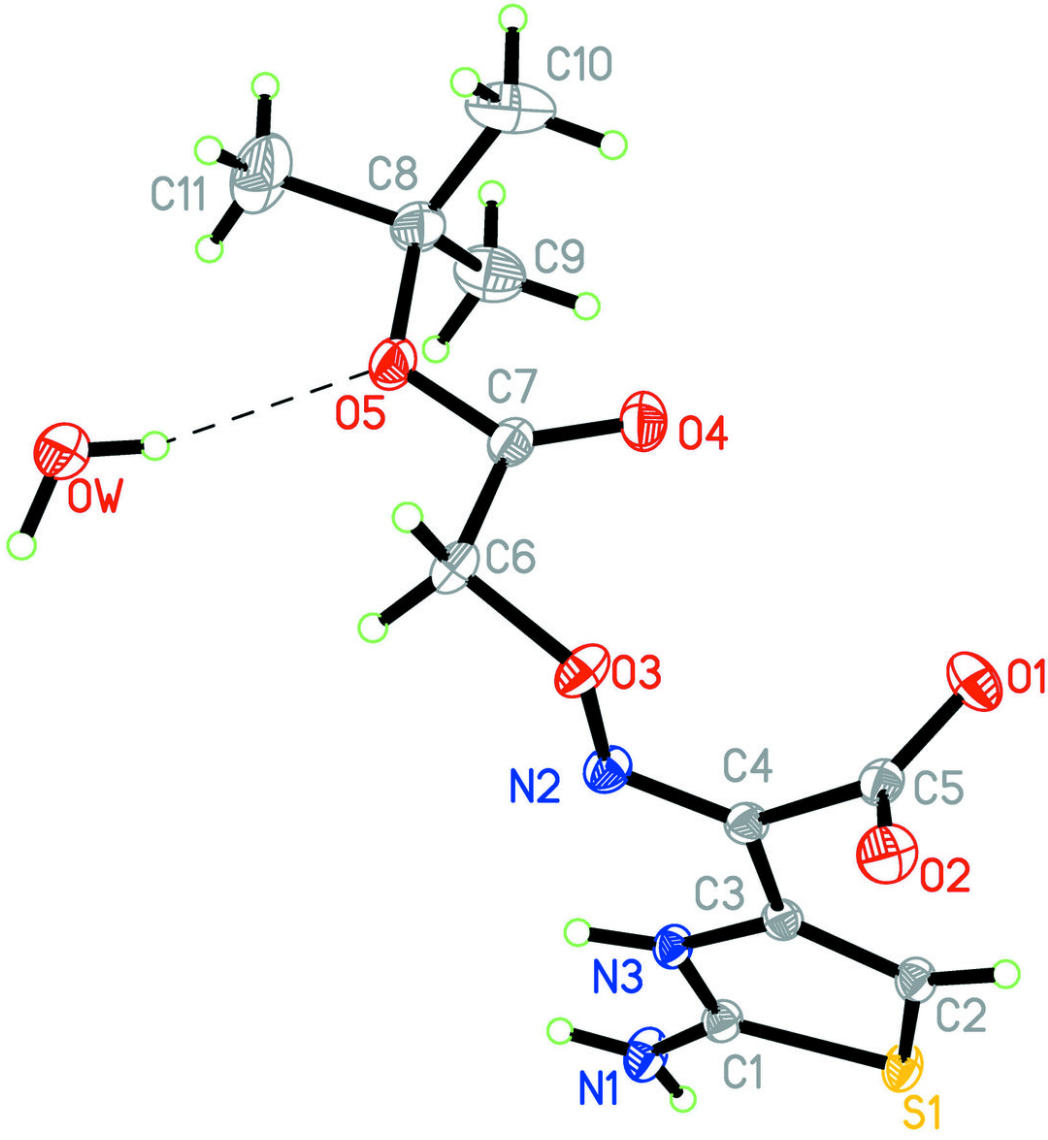


Fig. 2