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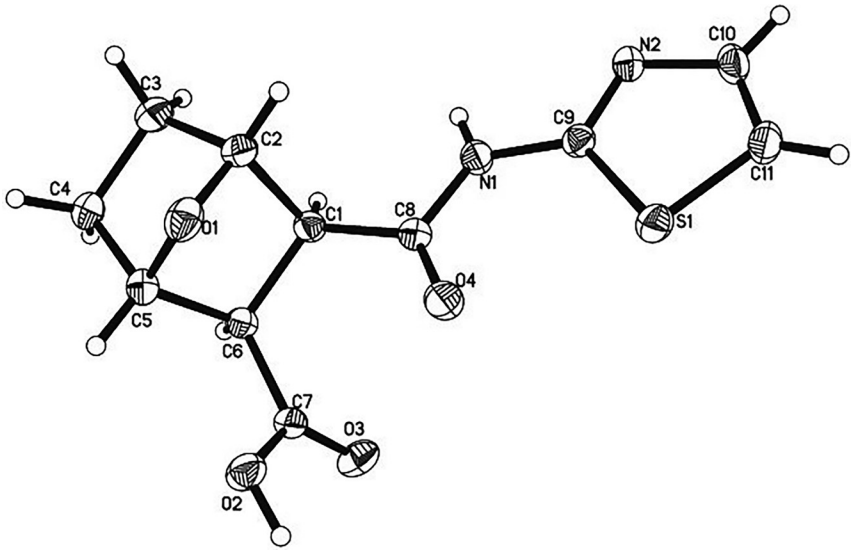
Crystal structure of 3-(thiazol-2-ylcarbamoyl)-7-oxabicyclo[2.2.1]heptane-2-carboxylic acid, $C_{11}H_{12}N_2O_4S$



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Abstract

$C_{11}H_{12}N_2O_4S_1$, orthorhombic, *Pbca* (no. 61), a = 11.08447(14) Å, b = 10.71473(16) Å, c = 19.5463(3) Å, V = 2321.46(6) Å^3, Z = 8, $R_{gt}(F)$ = 0.0450, $wR_{ref}(F^2)$ = 0.1275, T = 293(2) K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.14 × 0.11 × 0.10 mm
Wavelength:	Cu *Kα* radiation (1.54184 Å)
μ:	2.59 mm^{-1}
Diffractometer, scan mode:	Xcalibur, ω
θ_{max}, completeness:	70.4°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7851, 2195, 0.029
Criterion for I_{obs}, $N(hkl)_{gt}$:	1928
$N(param)_{refined}$:	171
Programs:	CrysAlis^{Pro} [1], Olex2 [2], Shelx [3, 4]

1 Source of materials

The title compound was prepared by the reaction of norcantharidin (6 mmol) and 2-aminothiazole (6 mmol) in the presence of 4-dimethylaminopyridine (0.01 mmol) as a catalyst, acetonitrile (20.0 mL) as a solvent. The mixture was stirred for 6 h at room temperature, providing precipitation, which was filtered, and then was washed with ethanol to give 3-(thiazol-2-ylcarbamoyl)-7-oxabicyclo[2.2.1] heptane-2-carboxylic acid in 85 % yield as white solid. The compound was crystallized as colourless crystals by slow evaporation from methanol at room temperature.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
S1	0.82681 (4)	0.75443 (4)	0.14377 (3)	0.0418 (2)
N1	0.68148 (16)	0.57262 (16)	0.19601 (9)	0.0362 (4)
H1	0.626 (3)	0.532 (2)	0.1872 (13)	0.039 (6)*
O1	0.63277 (14)	0.58903 (14)	0.40260 (8)	0.0439 (4)
C1	0.68490 (16)	0.47505 (17)	0.30552 (10)	0.0317 (4)
H1A	0.6565	0.4026	0.2795	0.038*
O2	0.91842 (13)	0.55123 (16)	0.41963 (8)	0.0458 (4)
H2	0.996 (3)	0.579 (3)	0.4180 (17)	0.075 (10)*
C2	0.57695 (18)	0.5376 (2)	0.34308 (12)	0.0423 (5)
H2A	0.5346	0.5997	0.3152	0.051*
N2	0.64422 (15)	0.64999 (16)	0.08639 (8)	0.0371 (4)
O3	0.96487 (13)	0.43876 (14)	0.32747 (8)	0.0449 (4)
C3	0.4941 (2)	0.4366 (3)	0.37185 (13)	0.0541 (6)
H3A	0.4784	0.3716	0.3385	0.065*
H3B	0.4181	0.4711	0.3876	0.065*
C4	0.57141 (19)	0.3879 (2)	0.43193 (11)	0.0430 (5)
H4A	0.5300	0.3976	0.4753	0.052*
H4B	0.5931	0.3009	0.4257	0.052*
O4	0.82128 (13)	0.64150 (14)	0.27128 (8)	0.0458 (4)
C5	0.68199 (17)	0.47272 (18)	0.42747 (10)	0.0356 (4)
H5	0.7257	0.4809	0.4708	0.043*
C6	0.76199 (16)	0.43081 (16)	0.36748 (9)	0.0303 (4)
H6	0.7632	0.3394	0.3671	0.036*
C7	0.89195 (17)	0.47602 (17)	0.36857 (9)	0.0321 (4)
C8	0.73979 (17)	0.57005 (17)	0.25743 (10)	0.0326 (4)
C9	0.70795 (17)	0.65139 (18)	0.14272 (9)	0.0328 (4)
C10	0.6911 (2)	0.7350 (2)	0.04030 (11)	0.0420 (5)
H10	0.6572	0.7477	−0.0027	0.050*
C11	0.7883 (2)	0.7977 (2)	0.06161 (12)	0.0453 (5)
H11	0.8295	0.8567	0.0357	0.054*

2 Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

3 Comment

Thiazole is a five-membered heterocyclic ring containing nitrogen and sulfur, and has been an important source of new drugs [5, 6]. The structure of the title compound has been obtained as part of a study of these materials [7, 8]. The asymmetric unit of the title structure consists of one molecule of the title compound (cf. the Figure). The molecule consists of a oxabicycloheptane group (O1, C1–C6) and a thiazole group (S1, N2, C9–C11). In the structure, the oxabicycloheptane group (O1, C1–C6) assumes a basket conformation, which is similar to several reported structures [9–11]. The dihedral angle between the plane (C1–C2–C5–C6) and the plane (C2–C5–O1) is 57.3°. The atoms of thiazole ring are coplanar. Adjacent molecules are linked through O–H … N hydrogen bonds (with the bond length of 2.720(2) Å) and N–H … O hydrogen bonds (with the bond length of 2.834(2) Å). The torsion angles of C8–N1–C9–N2, C8–N1–C9–S1, C6–C1–C8–O4, C5–C6–C7–O2, C5–C6–C7–O3 are –177.63(18), 4.0(3), –22.4(3), –3.2(2) and 172.87(18)°, respectively.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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