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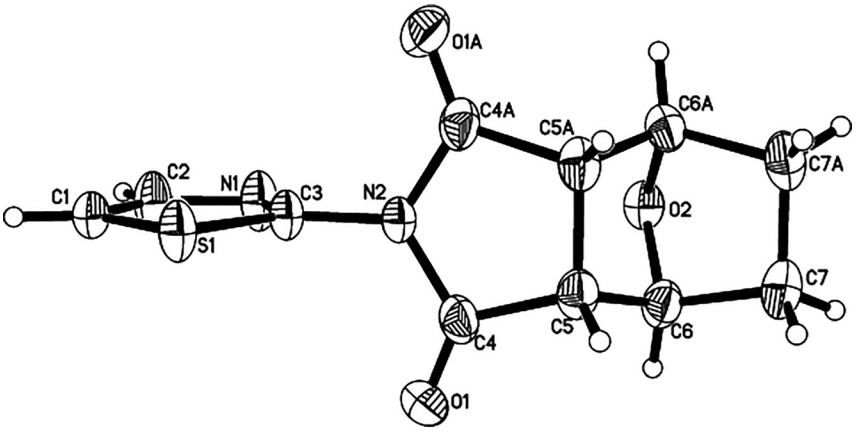
Crystal structure of 2-(thiazol-2-yl)hexahydro-1*H*-4,7-epoxyisoindole-1,3(2*H*)-dione, $C_{11}H_{10}N_2O_3S$



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Abstract

$C_{11}H_{10}N_2O_3S$, monoclinic, *Cm* (no. 8), a = 10.8483(7) Å, b = 9.3377(8) Å, c = 5.4086(3) Å, β = 95.398(6)°, V = 545.45(7) Å^3, Z = 2, $R_{gt}(F)$ = 0.0576, $wR_{ref}(F^2)$ = 0.1429, T = 293(2) K.

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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 needle
Size:	0.16 × 0.09 × 0.07 mm
Wavelength:	Cu *K*α radiation (1.54184 Å)
μ:	2.65 mm^{-1}
Diffractometer, scan mode:	Xcalibur, ω
θ_{max}, completeness:	70.2°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	2000, 756, 0.056
Criterion for I_{obs}, $N(hkl)_{gt}$:	620
$N(param)_{refined}$:	89
Programs:	Bruker [1], Olex2 [2], Shelx [3]

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 reaction mixture was stirred for 6 h at room temperature, providing precipitation, which was filtered, and then was washed with ethanol to give the intermediate 3-(thiazol-2-ylcarbamoyl)-7-oxabicyclo[2.2.1]heptane-2-carboxylic acid. To a stirred solution of 3-(thiazol-2-ylcarbamoyl)-7-oxabicyclo[2.2.1] heptane-2-carboxylic acid (5 mmol) in acetonitrile (10 mL) at 0 °C under Ar was added EDCI (5 mmol). The reaction mixture was heated at 80 °C for 5 h. After cooling to room temperature, excess amount of acetonitrile was removed under reduced pressure. The residue was purified by chromatography to give the target product in 85 % yield as white solid. The compound was crystallised by slow evaporation from a mixture solution of ethyl acetate and hexane at room temperature.

2 Experimental details

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

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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	0.3452 (3)	0.5000	0.0003 (7)	0.0576 (8)
O1	0.5071 (7)	0.7426 (6)	0.4833 (14)	0.0819 (19)
O2	0.6417 (7)	0.5000	0.9996 (12)	0.0498 (17)
N1	0.2634 (8)	0.5000	0.4270 (14)	0.063 (3)
N2	0.4838 (9)	0.5000	0.4443 (13)	0.0445 (19)
C1	0.1883 (10)	0.5000	0.0150 (17)	0.050 (2)
H1	0.1294	0.5000	−0.1215	0.060*
C2	0.1628 (9)	0.5000	0.2561 (17)	0.062 (3)
H2	0.0822	0.5000	0.3013	0.075*
C3	0.3617 (9)	0.5000	0.3198 (15)	0.047 (2)
C4	0.5493 (5)	0.6263 (9)	0.5068 (11)	0.0543 (19)
C5	0.6806 (5)	0.5823 (8)	0.6116 (8)	0.0520 (17)
H5	0.7445	0.6221	0.5150	0.062*
C6	0.7050 (5)	0.6161 (8)	0.8903 (8)	0.0558 (18)
H6	0.6784	0.7117	0.9377	0.067*
C7	0.8421 (5)	0.5825 (8)	0.9673 (14)	0.067 (2)
H7A	0.8683	0.6204	1.1308	0.081*
H7B	0.8952	0.6204	0.8483	0.081*

3 Comment

Norcantharidin was identified quite some time ago as a crucial building block for numerous chemotherapeutic agents [4, 5]. Many strategies have been developed to synthesize such molecules [6, 7]. Herein, we firstly report a new norcantharidin derivative obtained.

The molecule is located on a miror plane and thus the asymmetric unit contains one half (see the Figure). The molecule consists of a oxabicycloheptane group (O2, C5–C7, C5A–C7A), a lactam group (O1, O1A, N2, C4–C5, C4A–C5A) and a thiazole group (N1, S1, C1–C3). In the structure, the oxabicycloheptane group (O1, C1–C6) assumes a basket conformation. The atoms of thiazole ring are coplanar. The torsion angles of C4–N2–C3–N1, O1–C4–C5–C6, O1A–C4A–C5A–C6A are −91.0(7)°, 66.8(10)° and 176.7(8)°, respectively. Bond lengths and angles are all in the expected ranges [8].

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

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