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Crystal structure of 5-((2,3-dihydrobenzo[*b*][1,4]dioxin-6-yl)methylene)-thiazolidine-2,4-dione, $C_{12}H_9NO_4S$

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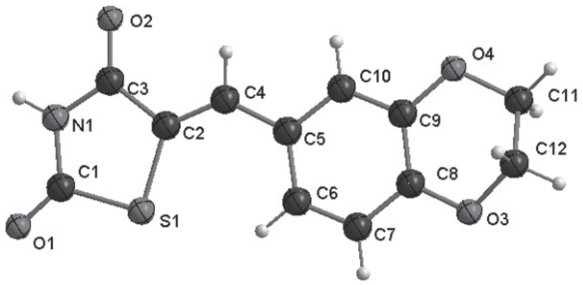

Abstract

$C_{12}H_9NO_4S$, triclinic, $P\overline{1}$ (no. 2), a = 3.9201(5) Å, b = 11.836(2) Å, c = 13.105(2) Å, α = 67.415(4)°, β = 83.375(4)°, γ = 82.156(4)°, V = 554.8 Å^3, Z = 2, $R_{gt}(F)$ = 0.0401, $wR_{ref}(F^2)$ = 0.0999, T = 293 K.

Table 1. Data collection and handling.

Crystal:	yellow blocks, size 0.18×0.20×0.21 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.97 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, ω
$2\theta_{max}$:	55.12°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5775, 2556
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2002
$N(param)_{refined}$:	163
Programs:	SHELX [10]

Source of material

2,3-Dihydrobenzo[*b*][1,4]dioxine-6-carbaldehyde (0.20 g, 1 mmol) and thiazolidine-2,4-dione (0.15 g, 1 mmol) were mixed in ethanol (20 mL) and refluxed for 10 hours under the catalytic impact of piperidine. After filtration and drying, the title compound was obtained. Then, 5 mg of the title compound was dissolved in 5 mL *N,N*-dimethyl formamide, and the solution was kept at room temperature for 7 d. Yellow block-shaped single crystals were obtained by slow evaporation.

Experimental details

Hydrogen atoms were positioned geometrically and refined using a riding model, with C–H = 0.93-0.97 Å, and with U_{iso}(H) set to 1.4 U_{eq} (C) and 1.6 U_{eq} (O).

Discussion

As we know, compounds conntaining the 1,4-benzodioxan structure exhibited strong biological activities, and are found widely spread in nature as well [1, 2]. Especially, the potential anticancer activities have received significant attention in chemical, medicinal and pharmaceutical research [3–5]. Previous studies has shown that the thiazolidine-2,4-dione derivatives were also associated with various biological activities [6–8] and have been recognized as new potential anticancer agents [9]. According to the pharmacological applications, we aimed to combine the two moieties together to strengthen their biological activities. All geometric parameters in the title structure are in the expected ranges. There are two intramolecular C–H···O and C–H···S hydrogen bonds in this molecule. The dihedral angle between the five heterocycle ring (C1, S1, C2, C3, N1) and benzene rings (C5, C6, C7, C8, C9, C10) is 4.5°.

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(3)	2*i*	0.7314	0.9539	0.6587	0.042
H(5)	2*i*	0.9104	0.7540	0.7648	0.043
H(7A)	2*i*	0.4525	0.6370	1.2060	0.049
H(6B)	2*i*	0.7960	0.6901	1.1430	0.049
H(4)	2*i*	0.2297	0.9975	0.9279	0.037
H(2)	2*i*	0.1767	1.1595	0.7638	0.038
H(9A)	2*i*	0.7320	0.5083	1.1201	0.049
H(8B)	2*i*	0.3799	0.5670	1.0666	0.049
H(1)	2*i*	0.2233	1.4615	0.4311	0.047

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(1)	2*i*	0.6235(1)	1.16986(4)	0.50543(4)	0.0468(3)	0.0323(2)	0.0343(2)	0.0082(2)	0.0027(2)	–0.0111(2)
C(8)	2*i*	0.6756(4)	0.7547(1)	0.9099(1)	0.0324(9)	0.0243(8)	0.0320(8)	0.0008(7)	–0.0041(7)	–0.0093(7)
C(6)	2*i*	0.6647(4)	0.9226(2)	0.7340(1)	0.044(1)	0.0326(9)	0.0266(8)	0.0029(8)	0.0018(7)	–0.0108(7)
C(7)	2*i*	0.7719(5)	0.8030(2)	0.7975(1)	0.042(1)	0.0301(9)	0.0336(9)	0.0057(7)	0.0019(7)	–0.0152(7)
O(4)	2*i*	0.3756(3)	0.7863(1)	1.07041(9)	0.0524(8)	0.0320(6)	0.0275(6)	0.0034(5)	0.0058(5)	–0.0074(5)
O(3)	2*i*	0.7879(3)	0.6350(1)	0.9687(1)	0.0499(8)	0.0260(6)	0.0337(7)	0.0066(5)	0.0009(5)	–0.0073(5)
C(11)	2*i*	0.5692(5)	0.6733(2)	1.1332(1)	0.048(1)	0.0378(9)	0.0293(9)	0.0014(8)	–0.0013(8)	–0.0051(7)
C(10)	2*i*	0.3659(4)	0.9484(1)	0.8950(1)	0.0326(9)	0.0282(8)	0.0315(8)	0.0015(7)	0.0019(7)	–0.0139(7)
C(5)	2*i*	0.4556(4)	0.9978(1)	0.7815(1)	0.0332(9)	0.0246(8)	0.0307(8)	0.0002(7)	–0.0020(7)	–0.0098(7)
C(4)	2*i*	0.3232(4)	1.1238(2)	0.7211(1)	0.0364(9)	0.0277(8)	0.0316(8)	0.0024(7)	–0.0019(7)	–0.0128(7)
C(12)	2*i*	0.6066(5)	0.5847(2)	1.0755(2)	0.047(1)	0.0289(9)	0.038(1)	–0.0009(8)	0.0015(8)	–0.0042(7)
C(9)	2*i*	0.4736(4)	0.8289(1)	0.9594(1)	0.0315(9)	0.0288(8)	0.0270(8)	–0.0031(7)	0.0005(6)	–0.0109(6)
O(2)	2*i*	0.0044(4)	1.3714(1)	0.6297(1)	0.0648(9)	0.0331(7)	0.0344(7)	0.0150(6)	0.0059(6)	–0.0088(5)
N(1)	2*i*	0.2998(4)	1.3860(1)	0.4653(1)	0.0510(9)	0.0260(7)	0.0311(7)	0.0074(6)	0.0003(7)	–0.0061(6)
C(3)	2*i*	0.2048(4)	1.3247(2)	0.5746(1)	0.039(1)	0.0276(8)	0.0304(8)	0.0023(7)	–0.0024(7)	–0.0094(7)
C(2)	2*i*	0.3728(4)	1.1983(2)	0.6150(1)	0.0339(9)	0.0281(8)	0.0322(8)	0.0025(7)	–0.0031(7)	–0.0128(7)
O(1)	2*i*	0.6324(4)	1.3642(1)	0.3175(1)	0.086(1)	0.0479(8)	0.0344(7)	0.0095(8)	0.0164(7)	–0.0061(6)
C(1)	2*i*	0.5219(5)	1.3229(2)	0.4120(2)	0.046(1)	0.0347(9)	0.0322(9)	0.0046(8)	0.0013(8)	–0.0095(7)

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