Crystal structure of 3-(indole-2-carbonyl)-2-thiono-thiazolidine, $C_{12}H_{10}N_2OS_2$

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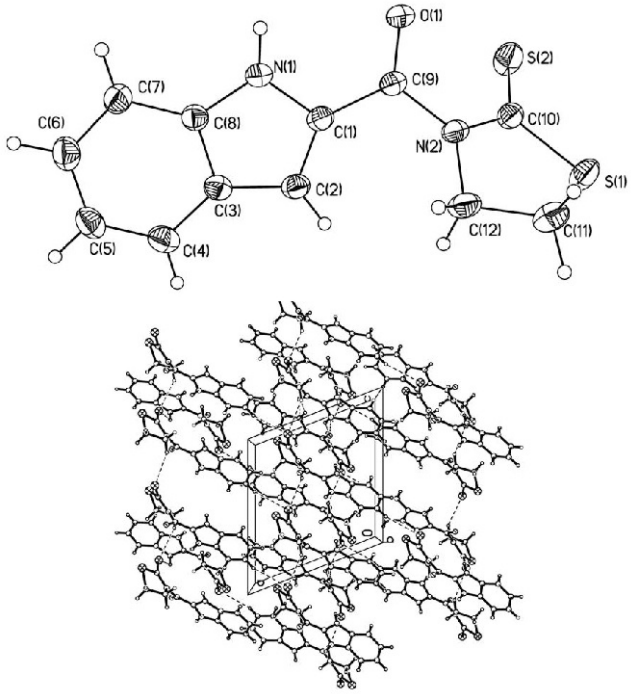
Received March 28, 2012, accepted June 12, 2012, available online August 09, 2012, CCDC no. 1267/3813



Abstract

$C_{12}H_{10}N_2Os_2$, monoclinic, $P2_1/c$ (no. 14), a = 10.572(2) Å, b = 10.449(2) Å, c = 11.348(2) Å, β = 111.202(2)°, V = 1168.7 Å^3, Z = 4, $R_{gt}(F)$ = 0.0355, $wR_{ref}(F^2)$ = 0.0967, T = 296 K.

Table 1. Data collection and handling.

Crystal:	yellow blocks, size 0.10×0.15×0.32 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	4.38 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8732, 2172
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1671
$N(param)_{refined}$:	154
Programs:	SHELX [4]

Source of material

To a solution of 1,3-thiazolidine-2-thione (5mmol) in 10 ml of anhydrous CH_3CN, an equimolar amount of indole-2-formylchloride in the presence of a catalytic amount of triethylamine was added. The reaction mixture was stirred for 20h until thin layer chromatography (TLC) indicated the end of reaction. Then, the precipitate of 3-(indole-2-carbonyl)-2-thiono-thiazolidine was collected by filtration and dried in air. Yellow single crystals suitable for *X*-ray diffraction were grown from a mixture of methylene chloride and petroleum (2:1) over a period of two weeks (yield 73%).

Discussion

There are numerous biologically active molecules with five-membered rings, containing two heteroatoms [1]. Compounds containing the 1,3-thiazolidine-2-thioneone ring have been shown a wide range of pharmacological activities [2,3]. Thus we envisioned that the 1,3-thiazolidine-2-thioneone derivative bearing indole moiety might have high biological activity.

The title compound, $C_{12}H_{10}N_2OS_2$, which crystallized in the monoclinic $P2_1/c$ space group, interact with neigboring molecules via weak intermolecular N–H···O and C–H···S hydrogen bonds. The bond lengths and angles of these hydrogen bonds are in the range of 2.945(2)-3.597(3) Å and 123 - 141°, respectively. These hydrogen bonds, albeit rather weak, link the title compound into a three-dimensional supramolecular structure (figure, bottom).

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

Atom	Site	x	y	z	U_{iso}
H(2)	4*e*	0.6567	0.383	0.3812	0.052
H(4)	4*e*	0.9378	0.4174	0.5585	0.062
H(5)	4*e*	1.0899	0.3387	0.7439	0.066
H(6)	4*e*	1.0331	0.1709	0.8479	0.066
H(7)	4*e*	0.8232	0.0745	0.7674	0.057
H(11A)	4*e*	0.2595	0.5866	0.1968	0.071
H(11B)	4*e*	0.1813	0.4837	0.2465	0.071
H(12A)	4*e*	0.4571	0.4772	0.2731	0.063
H(12B)	4*e*	0.3975	0.4503	0.3795	0.063
H(1)	4*e*	0.5792	0.0937	0.5667	0.049

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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}
C(1)	4*e*	0.5692(2)	0.2330(2)	0.4416(2)	0.043(1)	0.033(1)	0.039(1)	–0.0002(9)	0.0148(9)	0.0026(8)
C(2)	4*e*	0.6663(2)	0.3195(2)	0.4413(2)	0.050(1)	0.037(1)	0.044(1)	–0.003(1)	0.018(1)	0.0041(9)
C(3)	4*e*	0.7835(2)	0.2952(2)	0.5488(2)	0.042(1)	0.036(1)	0.046(1)	–0.0025(9)	0.018(1)	–0.0040(9)
C(4)	4*e*	0.9137(2)	0.3499(2)	0.5996(2)	0.047(1)	0.047(1)	0.064(2)	–0.008(1)	0.023(1)	–0.004(1)
C(5)	4*e*	1.0038(2)	0.3026(2)	0.7096(2)	0.037(1)	0.057(2)	0.065(2)	–0.003(1)	0.011(1)	–0.011(1)
C(6)	4*e*	0.9694(2)	0.2004(2)	0.7725(2)	0.047(1)	0.056(2)	0.053(1)	0.008(1)	0.006(1)	–0.004(1)
C(7)	4*e*	0.8450(2)	0.1430(2)	0.7259(2)	0.050(1)	0.042(1)	0.047(1)	0.004(1)	0.012(1)	0.002(1)
C(8)	4*e*	0.7521(2)	0.1916(2)	0.6136(2)	0.041(1)	0.035(1)	0.041(1)	0.0000(9)	0.0131(9)	–0.0034(9)
C(9)	4*e*	0.4372(2)	0.2030(2)	0.3476(2)	0.044(1)	0.033(1)	0.041(1)	–0.0010(9)	0.015(1)	0.0045(9)
C(10)	4*e*	0.2961(2)	0.2753(2)	0.1351(2)	0.044(1)	0.040(1)	0.043(1)	0.002(1)	0.016(1)	0.0044(9)
C(11)	4*e*	0.2498(3)	0.4958(2)	0.2092(2)	0.076(2)	0.038(1)	0.064(2)	0.006(1)	0.027(1)	0.001(1)
C(12)	4*e*	0.3827(3)	0.4381(2)	0.2908(2)	0.071(2)	0.031(1)	0.051(1)	–0.005(1)	0.016(1)	–0.000(1)
N(1)	4*e*	0.6217(2)	0.1551(2)	0.5467(2)	0.043(1)	0.0339(9)	0.043(1)	–0.0054(8)	0.0119(8)	0.0056(8)
N(2)	4*e*	0.3730(2)	0.3002(2)	0.2592(2)	0.046(1)	0.0299(9)	0.0401(9)	–0.0036(7)	0.0117(8)	0.0033(7)
O(1)	4*e*	0.3848(2)	0.0985(1)	0.3424(2)	0.056(1)	0.0376(9)	0.057(1)	–0.0128(7)	0.0050(8)	0.0123(7)
S(1)	4*e*	0.20620(7)	0.40990(6)	0.06220(6)	0.0753(5)	0.0458(4)	0.0511(4)	0.0153(3)	0.0109(3)	0.0095(3)
S(2)	4*e*	0.29158(7)	0.14393(6)	0.05758(6)	0.0765(5)	0.0485(4)	0.0532(4)	0.0106(3)	0.0069(3)	–0.0099(3)

Acknowledgments. The authors thank Henan University of Science and Technology for supporting this work.

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