Crystal structure of (*Z*)-5-(3-methylbenzylidene)-2-thioxothiazolidin-4-one, $C_{13}H_{15}NO_2S_3$

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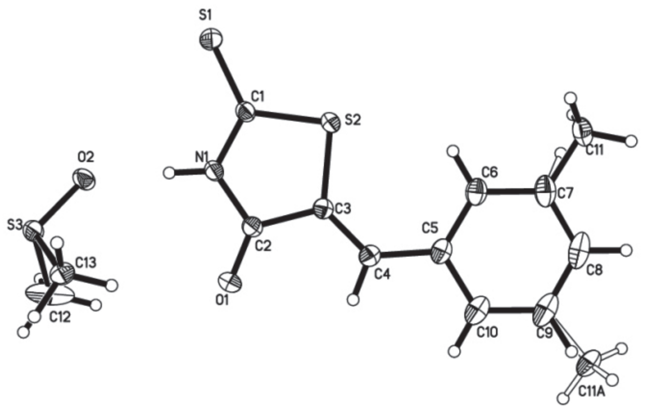

Abstract

$C_{13}H_{15}NO_2S_3$, triclinic, $P\overline{1}$ (no. 2), $a = 7.2618(5)$ Å, $b = 8.3190(6)$ Å, $c = 13.2073(10)$ Å, $\alpha = 93.117(2)°$, $\beta = 97.485(2)°$, $\gamma = 109.523(2)°$, $V = 741.5$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0573$, $wR_{ref}(F^2) = 0.1646$, $T = 150$ K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.234×0.323×0.532 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	4.96 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II D8 venture, φ and ω
$2\theta_{max}$:	72.94°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	44940, 7206
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4911
$N(param)_{refined}$:	188
Programs:	SHELX [6]

Source of material

The reagents and solvents used in this study are commercially available. The synthesis of (*Z*)-5-(3-methylbenzylidene)-2-thioxothiazolidin-4-one was carried out by reported our procedure [1]. A mixture of 3-methylbenzaldehyde (3 mmol, 360 mg), 2-thioxothiazolidin-4-one (3 mmol, 400 mg), and Et_2NH (3 mmol, 310 µl) in 3ml of degassed H_2O was stirred at room temperature for 3–5 hours until *TLC* showed complete disappearance of the reactants. Water (3 ml) was then added into the reaction mixture, then extracted with DCM/EtOH (3×50 ml) and the organic phase washed with 10%HCl (2×50 ml), followed by brine (2×50 ml), and dried over$MgSO_4$, and filtered off and evaporated to afford the desired product. The compound was obtained as colourless crystals (DMSO). **M. p.** 175 °C. **^{1}H-NMR** (400 MHz, $CDCl_3$): δ 7.57–7.32 (m, 6H, NH & CH & Ph), 2.37 (s, 3H, CH_3); **^{13}C-NMR** (100 MHz, $CDCl_3$): δ 196.3, 169.9, 139.4, 133.5, 132.3, 132.0, 131.5, 130.0, 129.9, 128.2, 128.1, 127.7, 125.8, 21.5.

Discussion

The rhodanine (2-thioxothiazolidin-4-one) ring system is a core structure in various synthetic pharmaceuticals displaying diverse pharmacological properties such as antiinflammatory, antiviral, anti-proliferative, anticonvulsant, anti-diabetic, anti-tubercular, cardiovascular, anti-hyperlipidemic, antifungal and antibacterial, properties. Several such derivatives as etozoline (antihypertensive), pioglitazone (hypoglycemic), ralitoline (anticonvulsant), and thiazolidomycin (activity against Streptomyces species), based on this pharmacophore are already on the market [1–6]. The title structure is composed of a planar thioxothiazolidin-4-one ring (C1/N1/C2/C3-S2) having a 3-methylbenzylidene substituent (C4/C5-C10) attached to C3. The dihedral angle between these two planes is 2.37(2)°. The methyl group C11 is disordered over two positions with an occupancy ratio of 0.67:0.33. The asymmetrical unit involves a dimethtyl sulphoxide (DMSO) solvate molecule (Fig.) which plays an important role in the stabilization of the crystal structure.

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

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(4A)	2*i*		0.6898	0.6402	0.3810	0.028
H(6A)	2*i*		0.8086	0.4157	0.5933	0.035
H(8A)	2*i*		1.0150	0.8394	0.7971	0.051
H(10A)	2*i*		0.8103	0.8739	0.5032	0.039
H(11A)	2*i*	0.645	1.0156	0.5973	0.8531	0.052
H(11B)	2*i*	0.645	1.0520	0.4634	0.7734	0.052
H(11C)	2*i*	0.645	0.8349	0.4311	0.8006	0.052
H(9A)	2*i*	0.645	0.9472	1.0044	0.6703	0.041
H(11D)	2*i*	0.355	1.0197	1.0707	0.7664	0.051
H(11E)	2*i*	0.355	0.8426	1.0842	0.6854	0.051
H(11F)	2*i*	0.355	1.0599	1.1311	0.6559	0.051
H(7A)	2*i*	0.355	0.9479	0.5478	0.7620	0.041
H(12A)	2*i*		0.0278	0.1780	–0.0581	0.103
H(12B)	2*i*		0.1876	0.3193	–0.1092	0.103
H(12C)	2*i*		0.2044	0.3267	0.0131	0.103
H(13A)	2*i*		0.6555	0.2387	–0.0286	0.045
H(13B)	2*i*		0.5664	0.3598	0.0323	0.045
H(13C)	2*i*		0.5554	0.3572	–0.0896	0.045
H(1N1)	2*i*		0.435(4)	0.133(4)	0.177(2)	0.037(7)

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

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(1)	2*i*		0.4928(1)	–0.12326(6)	0.29921(4)	0.0546(3)	0.0205(2)	0.0270(2)	0.0067(2)	0.0030(2)	0.0046(2)
S(2)	2*i*		0.65360(7)	0.20867(6)	0.43139(3)	0.0245(2)	0.0242(2)	0.0194(2)	0.0088(2)	0.0004(1)	0.0034(1)
O(1)	2*i*		0.5372(2)	0.4588(2)	0.2116(1)	0.0425(8)	0.0271(7)	0.0258(6)	0.0135(6)	0.0008(6)	0.0080(5)
N(1)	2*i*		0.5064(2)	0.1833(2)	0.2416(1)	0.0265(7)	0.0221(6)	0.0189(6)	0.0050(5)	–0.0005(5)	0.0035(5)
C(1)	2*i*		0.5433(3)	0.0844(2)	0.3147(1)	0.0241(7)	0.0225(7)	0.0193(7)	0.0059(6)	0.0024(6)	0.0041(5)
C(2)	2*i*		0.5597(3)	0.3569(2)	0.2702(1)	0.0228(7)	0.0231(7)	0.0222(7)	0.0076(6)	0.0030(6)	0.0035(6)
C(3)	2*i*		0.6490(2)	0.3973(2)	0.3804(1)	0.0178(7)	0.0227(7)	0.0230(7)	0.0066(5)	0.0022(5)	0.0025(6)
C(4)	2*i*		0.7081(3)	0.5590(2)	0.4255(1)	0.0204(7)	0.0231(7)	0.0271(8)	0.0071(6)	0.0031(6)	0.0015(6)
C(5)	2*i*		0.7951(3)	0.6306(2)	0.5304(2)	0.0174(7)	0.0277(8)	0.0292(8)	0.0056(6)	0.0035(6)	–0.0036(6)
C(6)	2*i*		0.8367(3)	0.5352(3)	0.6084(2)	0.0213(8)	0.0337(9)	0.0282(8)	0.0047(7)	0.0031(6)	–0.0021(7)
C(7)	2*i*		0.9186(3)	0.6124(3)	0.7078(2)	0.0233(8)	0.048(1)	0.0258(8)	0.0059(8)	0.0040(7)	–0.0030(8)
C(8)	2*i*		0.9589(3)	0.7862(4)	0.7294(2)	0.028(1)	0.053(1)	0.037(1)	0.0054(9)	0.0027(8)	–0.019(1)
C(9)	2*i*		0.9188(3)	0.8837(3)	0.6540(2)	0.0256(9)	0.039(1)	0.053(1)	0.0079(8)	0.0040(9)	–0.019(1)
C(10)	2*i*		0.8376(3)	0.8066(3)	0.5550(2)	0.0205(8)	0.0313(9)	0.043(1)	0.0076(7)	0.0054(7)	–0.0090(8)
C(11)	2*i*	0.645	0.9584(5)	0.5187(5)	0.7901(2)	0.030(2)	0.042(2)	0.024(1)	0.004(1)	–0.002(1)	0.004(1)
C(11A)	2*i*	0.355	0.9636(9)	1.0555(7)	0.6934(5)	0.031(3)	0.024(2)	0.043(3)	0.007(2)	0.004(2)	–0.008(2)
S(3)	2*i*		0.31643(8)	0.11909(6)	–0.04890(4)	0.0386(3)	0.0202(2)	0.0226(2)	0.0002(2)	–0.0052(2)	0.0015(2)
O(2)	2*i*		0.2986(2)	0.0410(2)	0.0513(1)	0.0390(8)	0.0230(6)	0.0251(6)	–0.0005(5)	–0.0040(6)	0.0067(5)
C(12)	2*i*		0.1674(4)	0.2505(4)	–0.0510(4)	0.035(1)	0.049(2)	0.126(3)	0.013(1)	0.009(2)	0.049(2)
C(13)	2*i*		0.5496(3)	0.2876(2)	–0.0318(2)	0.0331(9)	0.0211(8)	0.0313(9)	0.0038(7)	0.0031(7)	0.0027(7)

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