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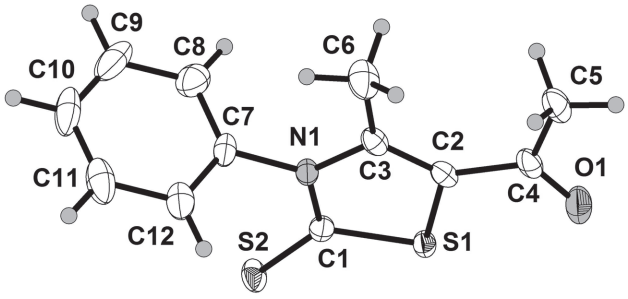
Crystal structure of 1-(2,3-dihydro-4-methyl-3-phenyl-2-thioxothiazol-5-yl)-1-ethanone, $C_{12}H_{11}NOS_2$



DOI 10.1515/ncrs-2016-0008
Received January 3, 2016; accepted March 21, 2016; available online April 21, 2016

Abstract

$C_{12}H_{11}NOS_2$, monoclinic, $P2_1/n$ (no. 14), $a = 12.4674$ (6) Å, $b = 6.7357$ (3) Å, $c = 14.0709$ (6) Å, $\beta = 98.189$ (2)°, $V = 1169.58$ (9) Å3, $Z = 4$, $R_{gt}(F) = 0.0316$, $wR_{ref}(F^2) = 0.0802$, $T = 100$.

CCDC no.: 1444405

Tables 1 and 2 contain details of the methods used and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The synthesis of 1-(2,3-dihydro-4-methyl-3-phenyl-2-thioxothiazol-5-yl)-1-ethanone was not carried out by our reported procedure [10–12]. 1-(2,3-dihydro-4-methyl-3-phenyl-2-thioxothiazol-5-yl)-1-ethanone was synthesis with

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Table 1: Data collection and handling.

Crystal:	Yellow blocks
	size 0.49 × 0.45 × 0.13
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	4.31
Diffractometer, scan mode:	Bruker Apex-II φ and ω
$2\theta_{max}$, completeness:	65,56°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	16903, 2049
R_{int}:	0.0554
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma\,(I_{obs})$, 1679
$N(param)_{refined}$:	147
Programs:	SHELX [8], Bruker programs [9]

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

Atom	x	y	z	U_{iso}*/U_{eq}
S1	0.71608 (4)	0.28622 (7)	0.84497 (3)	0.01236 (14)
S2	0.49533 (4)	0.28767 (8)	0.90468 (3)	0.01677 (15)
O1	0.91334 (11)	0.2919 (2)	0.76908 (10)	0.0222 (3)
N1	0.54533 (12)	0.2873 (2)	0.72320 (11)	0.0119 (3)
C1	0.57605 (15)	0.2869 (3)	0.82127 (13)	0.0123 (4)
C2	0.72826 (15)	0.2867 (3)	0.72273 (13)	0.0126 (4)
C3	0.62924 (15)	0.2865 (3)	0.66783 (13)	0.0137 (4)
C4	0.83983 (16)	0.2879 (3)	0.70054 (14)	0.0153 (4)
C5	0.86423 (17)	0.2856 (3)	0.59957 (15)	0.0201 (4)
H5A	0.9428	0.2785	0.6000	0.030*
H5B	0.8362	0.4071	0.5667	0.030*
H5C	0.8297	0.1699	0.5658	0.030*
C6	0.60242 (17)	0.2860 (4)	0.56090 (14)	0.0238 (5)
H6A	0.6445	0.3892	0.5339	0.036*
H6B	0.5249	0.3123	0.5427	0.036*
H6C	0.6202	0.1560	0.5360	0.036*
C7	0.43146 (15)	0.2935 (3)	0.68402 (13)	0.0150 (4)
C8	0.37913 (17)	0.1191 (3)	0.65377 (14)	0.0230 (5)
H8A	0.4172	-0.0034	0.6571	0.028*
C9	0.26913 (18)	0.1276 (4)	0.61828 (15)	0.0320 (6)
H9A	0.2315	0.0100	0.5960	0.038*
C10	0.21436 (18)	0.3068 (4)	0.61538 (15)	0.0310 (6)
H10A	0.1393	0.3114	0.5912	0.037*
C11	0.26791 (17)	0.4785 (4)	0.64726 (14)	0.0273 (5)
H11A	0.2296	0.6006	0.6455	0.033*
C12	0.37804 (16)	0.4732 (3)	0.68194 (13)	0.0201 (5)
H12A	0.4157	0.5909	0.7038	0.024*

a new and different way: to aniline (4 mmol) and 10 mL CS2 was slowly added 3-chloro-pentane-2,4-dione (4 mmol). The reaction mixture was stirred for 4–5 h. The residue was recrystallized from ethanol to form yellow crystals; yield (75%); m.p.: 175–177°C. ^{1}H-NMR 400 MHz, $CDCl_3$): : δ(ppm) 1.18 (s, CH_3), 2.35 (s, CH_3), 7.14–7.16 (m, 2 CH), 7.50–7.52 (m, 3 CH) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): d = 16.1 (CH_3), 30.2 (CH_3), 120.9 (C), 128.0 (2 CH), 130.1 (CH), 130.2 (2 CH), 137.3 (C—N), 147.3 (C), 188.1 (C), 190.5 (C) ppm.

Discussion

Compounds of thiazoline-2-thiones are important for photographic materials [1], and in synthesis of heterocyclic compounds [2–4]. Thiazoline-2-thiones derivatives are present in the heterocyclic units of many biomolecules and sulphur coordination compounds [5]. They are also powerful inhibitors of carbonic anhydrase activity [6, 7].

The molecules packing in the crystal structure is at least stabilized *via* one intermolecular non-classical hydrogen bond, of which O1 work as hydrogen bond acceptor and C12 work as hydrogen bond donor. The distance of the interaction between C12—H12A$\cdots$O1^{i} is 2.51 Å, and the angle is 150°. Symmetry codes: (i) $-x+3/2$, $y+1/2$, $-z+3/2$.

Acknowledgements: The authors extend their sincere appreciation to the Deanship of Scientific Research at king Saud University for its funding this Prolific Research group (PRG-1437-29).

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