

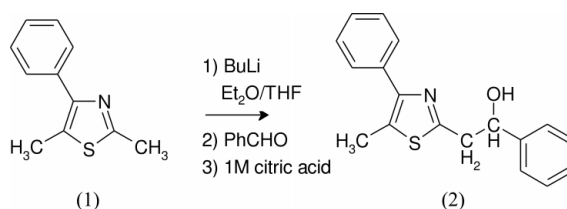
2-(5-Methyl-4-phenyl-1,3-thiazol-2-yl)-
1-phenylethanolVictor B. Rybakov,^{a*}
Antonina Yu. Liakina,^b
Inna S. Popova,^b
Andrey A. Formanovsky^b and
Leonid A. Aslanov^a^aDepartment of Chemistry, Moscow State University, 119992 Moscow, Russian Federation, and ^bM. M. Shemyakin and Yu. A. Ovchinnikov Institute of Bioorganic Chemistry, Russian Academy of Sciences, 117781 Moscow, Russian FederationCorrespondence e-mail:
rybakov@biocryst.phys.msu.su

Key indicators

Single-crystal X-ray study
 $T = 293\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$
 R factor = 0.040
 wR factor = 0.116
Data-to-parameter ratio = 15.4For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.The title compound, $\text{C}_{18}\text{H}_{17}\text{NOS}$, (2), is the product of substitution at the methyl group, in the 2-position of 2,5-dimethyl-4-phenylthiazole, with benzaldehyde. Molecules of (2) form extended chains through $\text{O}-\text{H}\cdots\text{N}$ hydrogen bonds.Received 28 July 2003
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Comment

The metallation of 2,5-dimethyl-4-phenylthiazole by butyllithium can generate anionic methyl groups at positions 2 and 5. The title structure, (2), is the product of metallation and the subsequent reaction of the anion with benzaldehyde, and was prepared according to the Scheme below.

The 5-membered heterocycle (S1/C2/N3/C4/C5) (Fig. 1) is nearly planar and there is conjugation between it and the attached phenyl ring (C6–C11), the dihedral angle being $6.83(11)^\circ$. An $\text{O13}-\text{H131}\cdots\text{N3}^i$ intermolecular hydrogen bond [$\text{H131}\cdots\text{N3}^i = 1.96(4)\text{ \AA}$, $\text{O13}\cdots\text{N3}^i = 2.831(2)\text{ \AA}$ and $\text{O13}-\text{H131}\cdots\text{N3}^i = 179(3)^\circ$; symmetry code: (i) $x, \frac{1}{2}-y, z - \frac{1}{2}$] links the molecules in the crystal into extended chains.

Experimental

Solvents were dried and distilled under an inert atmosphere. The starting thiazole, (1), was prepared from thioacetamide and 2-bromo-1-phenylpropanone using the procedure of Hantch (1889). The reaction was carried out under an inert atmosphere (dry Argon). The ^1H NMR spectrum of (2) was recorded on a Bruker AMX-400. The melting point was determined on a hot-stage Boëtius apparatus. BuLi in C_6H_{14} (6 ml, 1.6 M solution) was added to a solution of thiazole (1) (1 g, 5.3 mmol) in 20 ml $\text{Et}_2\text{O}/4\text{ ml C}_4\text{H}_8\text{O}$ for 10 min at 203 K (dry $\text{CO}_2/\text{C}_7\text{H}_8$). Benzaldehyde (0.67 g, 6 mmol) in 2 ml of $\text{C}_4\text{H}_8\text{O}$ was added dropwise to the reaction mixture after 30 min of stirring at 203 K. Then 10 ml of 1 M aqueous citric acid were added after 30 min of stirring and the reaction mixture was left to warm to room temperature; it was then washed with water and brine, dried over Na_2SO_4 and filtered. The solvent was evaporated *in vacuo* and 20 ml of C_6H_{14} was added to the resulting yellow oil; after 15 min of heating the mixture was cooled, yielding 0.5 g (60%) of a white precipitate. The product was recrystallized from $\text{CH}_2\text{Cl}_2/\text{C}_6\text{H}_{14}$, m.p. 363–365 K. ^1H NMR (CDCl_3 , 400 MHz, p.p.m.): 7.64 (d, 2H, Ph), 7.43 (t, 4H, Ph), 7.35 (t, 3H, Ph), 7.28 (d, 1H, Ph), 5.19 (dd, 1H, CH), 3.30 (dd, 1H, CH_2 , $J = 4.5\text{ MHz}$), 3.25 (dd, 1H, CH_2 , $J = 8.5\text{ MHz}$), 2.53 (s, 3H, CH_3).

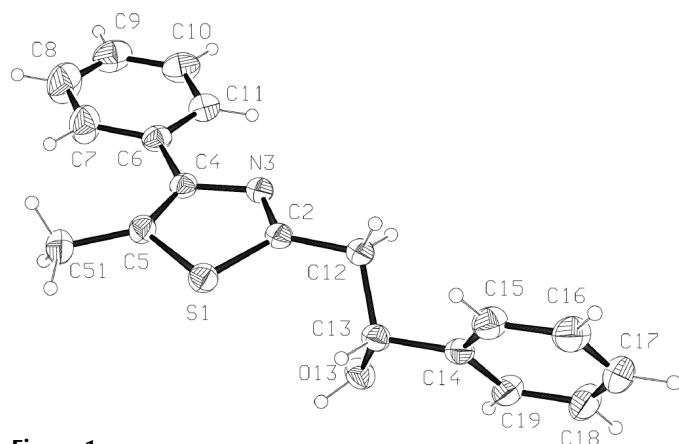


Figure 1
ORTEP-3 (Farrugia, 2003) plot of the molecule of the title compound. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as spheres of arbitrary radius.

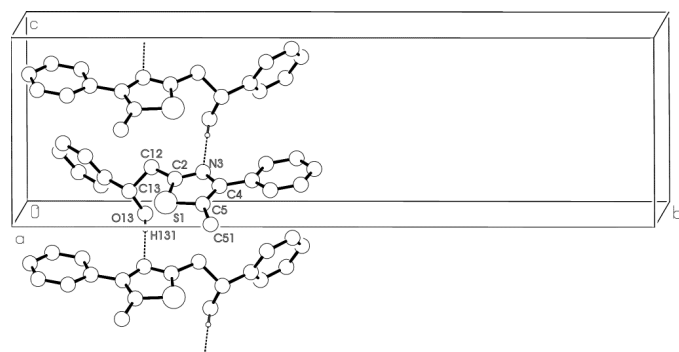


Figure 2
PLUTON97 (Spek, 1997) packing diagram, with the hydrogen-bonding scheme indicated by dashed lines.

Crystal data

$C_{18}H_{17}NOS$
 $M_r = 295.39$
 Monoclinic, $P2_1/c$
 $a = 6.2820$ (9) Å
 $b = 28.836$ (4) Å
 $c = 8.4583$ (13) Å
 $\beta = 97.560$ (10)°
 $V = 1518.9$ (4) Å³
 $Z = 4$

$D_x = 1.292$ Mg m⁻³
 Cu $K\alpha$ radiation
 Cell parameters from 25 reflections
 $\theta = 33\text{--}35^\circ$
 $\mu = 1.86$ mm⁻¹
 $T = 293$ (2) K
 Prism, colourless
 $0.30 \times 0.30 \times 0.30$ mm

Data collection

Enraf-Nonius CAD-4 diffractometer
 Non-profiled $\omega/2\theta$ scans
 Absorption correction: none
 3201 measured reflections
 2997 independent reflections
 2613 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.021$

$\theta_{max} = 74.9^\circ$
 $h = -7 \rightarrow 7$
 $k = 0 \rightarrow 36$
 $l = 0 \rightarrow 9$
 1 standard reflection every 226 reflections
 frequency: 49 min
 intensity decay: 2%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.116$
 $S = 1.05$
 2997 reflections
 195 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0712P)^2 + 0.2241P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 0.31$ e Å⁻³
 $\Delta\rho_{min} = -0.28$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

S1—C2	1.7126 (17)	C9—C10	1.375 (4)
S1—C5	1.7270 (16)	C10—C11	1.385 (3)
C2—N3	1.299 (2)	C12—C13	1.535 (2)
C2—C12	1.505 (2)	C13—O13	1.4203 (19)
N3—C4	1.395 (2)	C13—C14	1.517 (2)
C4—C5	1.375 (2)	O13—H131	0.87 (3)
C4—C6	1.481 (2)	C14—C19	1.385 (2)
C5—C51	1.497 (2)	C14—C15	1.388 (2)
C6—C7	1.387 (3)	C15—C16	1.389 (3)
C6—C11	1.395 (3)	C16—C17	1.373 (4)
C7—C8	1.386 (3)	C17—C18	1.375 (3)
C8—C9	1.373 (4)	C18—C19	1.387 (3)
C2—S1—C5	90.68 (8)	C8—C9—C10	118.8 (2)
N3—C2—C12	123.73 (15)	C9—C10—C11	120.6 (2)
N3—C2—S1	114.11 (12)	C10—C11—C6	121.2 (2)
C12—C2—S1	122.16 (12)	C2—C12—C13	112.99 (13)
C2—N3—C4	111.88 (14)	O13—C13—C14	111.21 (13)
C5—C4—N3	114.35 (14)	O13—C13—C12	108.40 (13)
C5—C4—C6	127.76 (15)	C14—C13—C12	110.66 (12)
N3—C4—C6	117.89 (15)	C13—O13—H131	107 (2)
C4—C5—C51	133.52 (16)	C19—C14—C15	118.74 (16)
C4—C5—S1	108.97 (12)	C19—C14—C13	121.07 (15)
C51—C5—S1	117.41 (13)	C15—C14—C13	120.19 (15)
C7—C6—C11	117.30 (17)	C14—C15—C16	120.40 (19)
C7—C6—C4	122.51 (17)	C17—C16—C15	120.3 (2)
C11—C6—C4	120.19 (16)	C16—C17—C18	119.68 (19)
C8—C7—C6	121.0 (2)	C17—C18—C19	120.4 (2)
C9—C8—C7	121.1 (2)	C14—C19—C18	120.43 (19)

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O13—H131 \cdots N3 ⁱ	0.87 (4)	1.96 (4)	2.831 (2)	179 (3)

Symmetry code: (i) $x, \frac{1}{2} - y, z - \frac{1}{2}$.

The hydroxyl H atom was refined isotropically to give an O—H bond length of 0.87 (3) Å. H atoms bonded to C atoms were included in calculated positions and refined in the riding-motion approximation (C—H = 0.93–0.98 Å), with $U_{iso} = 1.2U_{eq}$ (1.5 U_{eq} for methyl) of the carrier atom.

Data collection: CAD-4 EXPRESS (Enraf-Nonius, 1994); cell refinement: CAD-4 EXPRESS (Enraf-Nonius, 1994); data reduction: XCAD4 (Harms & Wocadlo, 1995); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 for Windows (Farrugia, 2003) and PLUTON97 (Spek, 1997); software used to prepare material for publication: WinGX publication routines (Farrugia, 2003).

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