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Crystal structure of 2-(1-methylethyl)-1,3-thiazolo[4,5-*b*]pyridine

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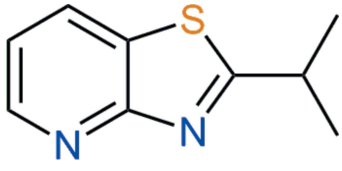
In the title molecule, $C_9H_{10}N_2S$, one of the methyl groups is almost co-planar with the thiazolopyridine rings with a deviation of 0.311 (3) Å from the least-squares plane of the thiazolopyridine group. In the crystal, weak C—H···N hydrogen-bonding interactions lead to the formation of chains along [011].

Keywords: crystal structure; thiazolopyridine; hydrogen bonding.

CCDC reference: 1056012

1. Related literature

For related compounds, see: Smith *et al.* (1994, 1995); El-Hiti (2003); Johnson *et al.* (2006); Thomae *et al.* (2008); Rao *et al.* (2009); Lee *et al.* (2010); Luo *et al.* (2015). For the X-ray crystal structures of related compounds, see: Yu *et al.* (2007); El-Hiti *et al.* (2014).



2. Experimental

2.1. Crystal data

$C_9H_{10}N_2S$
M_r = 178.25
Orthorhombic, $Pna2_1$
a = 9.6376 (2) Å
b = 10.1602 (2) Å
c = 8.9254 (2) Å
V = 873.98 (3) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 2.81 mm^{-1}
T = 150 K
0.23 × 0.20 × 0.14 mm

2.2. Data collection

Agilent SuperNova, Dual, Cu at zero, Atlas diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2014)
T_{min} = 0.897, T_{max} = 0.940
2848 measured reflections
1366 independent reflections
1351 reflections with $I > 2\sigma(I)$
R_{int} = 0.012

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.021
$wR(F^2)$ = 0.057
S = 1.08
1366 reflections
111 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···N1^{i}	0.95	2.51	3.391 (2)	153

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

Data collection: *CrysAlis PRO* (Agilent, 2014); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS2013* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *CHEMDRAW* Ultra (Cambridge Soft, 2001).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: ZS2329).

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Acta Cryst. (2015). E**71**, o272–o273 [doi:10.1107/S2056989015006039]

Crystal structure of 2-(1-methylethyl)-1,3-thiazolo[4,5-*b*]pyridine

Gamal A. El-Hiti, Keith Smith, Amany S. Hegazy, Saud A. Alanazi and Benson M. Kariuki

S1. Chemical context

Thiazolopyridines have been efficiently synthesized and in high yield using different synthetic procedures (Smith *et al.*, 1994, 1995; El-Hiti, 2003; Johnson *et al.*, 2006; Thomae *et al.*, 2008; Rao *et al.*, 2009; Lee *et al.*, 2010; Luo *et al.*, 2015). During our continuing research towards the development of novel synthetic routes for the production of heterocyclic compounds, we have synthesised the title compound 2-(methylethyl)-1,3-thiazolo[4,5-*b*]pyridine in high yield (Smith *et al*., 1995). The X-ray structures for related compounds have been reported (Yu *et al.*, 2007; El-Hiti *et al.*, 2014).

S2. Structural commentary

The asymmetric unit of the title compound consists of a single molecule of $C_9H_{10}N_2S$ (Fig. 1). In the molecule, one of the methyl groups is almost co-planar with the thiazolopyridine ring. The deviations from the least-squares plane of the thiazolopyridine group are 0.311 (3)Å and 1.269 (3)Å for C8 and C9 respectively, corresponding to torsion angles N1—C1—C7—C8 and N1—C1—C7—C9 of 169.47 (19) and -65.9 (3)°, respectively.

Weak C—H···N hydrogen-bonding interactions occur in the structure to form chains along [011] (Fig. 2, Table 1). No π–π interactions are observed in the crystal structure.

S3. Synthesis and crystallization

2-(1-Methylethyl)-1,3-thiazolo[4,5-*b*]pyridine was obtained in 98% yield from acid hydrolysis (HCl, 5 M) of 3-(diisopropylaminothiocarbonylthio)-2-(1-methylethylcarbonylamino)pyridine under reflux for 5 h (Smith *et al.*, 1995). Crystallization of the crude product from diethyl ether gave colourless crystals of the title compound. The spectroscopic and analytical data for the title compound were consistent with those reported previously (Smith *et al.*, 1995).

S4. Refinement details

H atoms were positioned geometrically and refined using a riding model with U_{iso}(H) constrained to be 1.2 times U_{eq} for the atom it is bonded to except for methyl groups where it was 1.5 times with free rotation about the C—C bond. Although not of relevance with this achiral compound, the absolute structure factor (Flack, 1983) was determined as 0.031 (11) for 434 Friedel pairs.

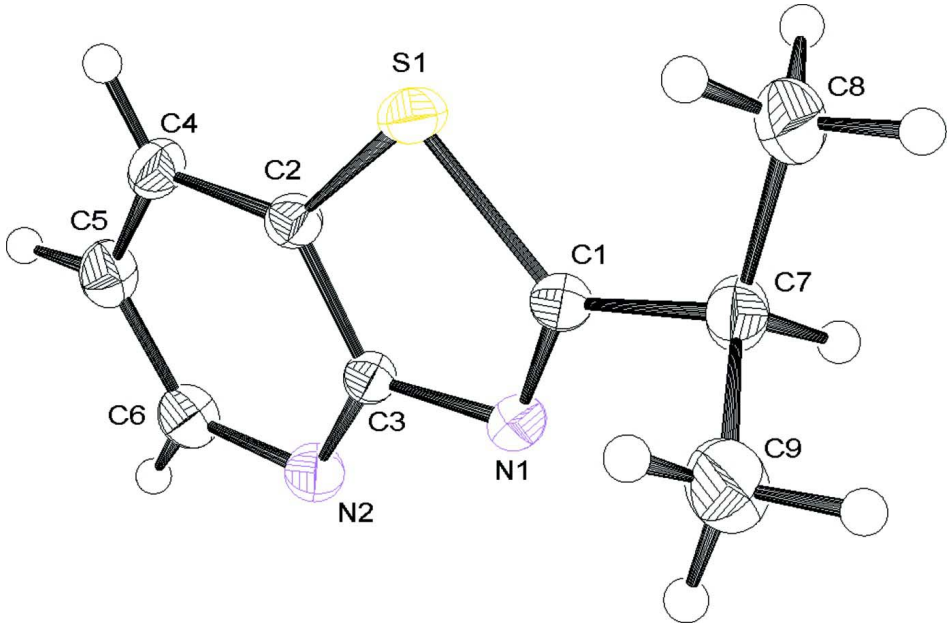

Figure 1

A molecule of $C_9H_{10}N_2S$ with atom labels and 50% probability displacement ellipsoids for non-hydrogen atoms.

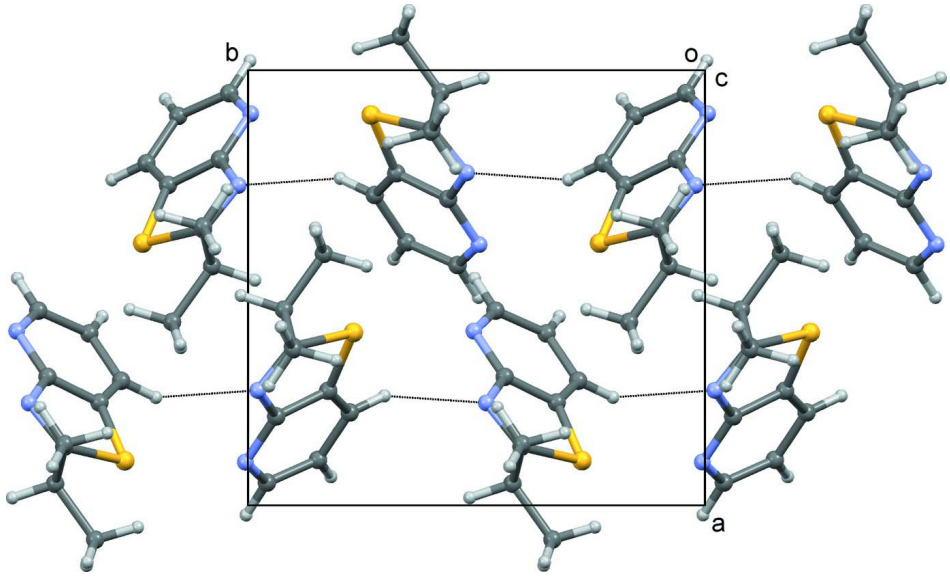

Figure 2

Crystal structure packing viewed down the *c* axis with C—H···N interactions shown as dotted lines.

2-(1-Methylethyl)-1,3-thiazolo[4,5-*b*]pyridine

Crystal data

$C_9H_{10}N_2S$
$M_r = 178.25$
Orthorhombic, $Pna2_1$
$a = 9.6376$ (2) Å
$b = 10.1602$ (2) Å
$c = 8.9254$ (2) Å
$V = 873.98$ (3) Å^3
$Z = 4$
$F(000) = 376$
$D_x = 1.355$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 2276 reflections

θ = 6.3–73.8°
μ = 2.81 mm^{-1}
T = 150 K

Block, colourless
0.23 × 0.20 × 0.14 mm

Data collection

Agilent SuperNova, Dual, Cu at zero, Atlas diffractometer
Radiation source: sealed X-ray tube, SuperNova (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.5082 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2014)

T_{min} = 0.897, T_{max} = 0.940
2848 measured reflections
1366 independent reflections
1351 reflections with $I > 2\sigma(I)$
R_{int} = 0.012
θ_{max} = 74.0°, θ_{min} = 6.3°
h = −11→10
k = −12→8
l = −10→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.021
$wR(F^2)$ = 0.057
S = 1.08
1366 reflections
111 parameters
1 restraint

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0375P)^2 + 0.1081P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
C1	0.13501 (18)	0.59856 (18)	0.8660 (2)	0.0227 (4)
C2	0.23657 (17)	0.67547 (17)	1.1015 (2)	0.0208 (3)
C3	0.29550 (18)	0.56187 (17)	1.0365 (2)	0.0203 (3)
C4	0.2865 (2)	0.72261 (17)	1.2365 (3)	0.0257 (4)
H4	0.2483	0.7985	1.2829	0.031*
C5	0.39534 (19)	0.6532 (2)	1.3007 (3)	0.0279 (4)
H5	0.4342	0.6813	1.3931	0.034*
C6	0.44760 (19)	0.54145 (19)	1.2283 (3)	0.0273 (4)
H6	0.5220	0.4959	1.2752	0.033*
C7	0.05003 (18)	0.57871 (19)	0.7259 (3)	0.0271 (4)
H7	0.0177	0.4852	0.7251	0.033*
C8	−0.0783 (2)	0.6663 (2)	0.7204 (3)	0.0365 (5)
H8A	−0.1375	0.6475	0.8070	0.055*
H8B	−0.1299	0.6488	0.6279	0.055*
H8C	−0.0499	0.7589	0.7227	0.055*
C9	0.1411 (2)	0.5992 (3)	0.5875 (3)	0.0421 (6)
H9A	0.1666	0.6923	0.5796	0.063*
H9B	0.0897	0.5727	0.4977	0.063*

H9C	0.2253	0.5457	0.5966	0.063*
N1	0.23563 (16)	0.52156 (16)	0.9037 (2)	0.0241 (3)
N2	0.40055 (14)	0.49434 (16)	1.0985 (2)	0.0254 (4)
S1	0.10273 (4)	0.73079 (4)	0.98822 (7)	0.02515 (14)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0205 (8)	0.0253 (8)	0.0224 (10)	−0.0013 (7)	0.0032 (7)	−0.0026 (8)
C2	0.0212 (8)	0.0210 (7)	0.0203 (9)	−0.0011 (6)	0.0033 (6)	−0.0004 (7)
C3	0.0199 (7)	0.0206 (7)	0.0204 (9)	−0.0026 (6)	0.0032 (6)	−0.0024 (7)
C4	0.0292 (9)	0.0258 (9)	0.0221 (9)	−0.0028 (7)	0.0018 (8)	−0.0061 (8)
C5	0.0302 (9)	0.0353 (11)	0.0184 (9)	−0.0058 (7)	−0.0024 (7)	0.0002 (9)
C6	0.0258 (8)	0.0303 (9)	0.0259 (9)	0.0005 (7)	−0.0035 (8)	0.0042 (8)
C7	0.0266 (8)	0.0293 (9)	0.0254 (9)	−0.0036 (7)	−0.0032 (8)	−0.0030 (8)
C8	0.0293 (9)	0.0428 (12)	0.0374 (13)	0.0037 (9)	−0.0127 (9)	−0.0071 (11)
C9	0.0351 (11)	0.0705 (16)	0.0207 (11)	−0.0072 (11)	−0.0024 (8)	−0.0025 (11)
N1	0.0231 (7)	0.0264 (8)	0.0229 (8)	0.0012 (6)	0.0001 (6)	−0.0067 (7)
N2	0.0238 (8)	0.0239 (7)	0.0284 (9)	0.0024 (6)	−0.0016 (6)	−0.0002 (7)
S1	0.0242 (2)	0.0266 (2)	0.0246 (2)	0.00703 (13)	−0.0007 (2)	−0.0048 (2)

Geometric parameters (Å, º)

C1—N1	1.291 (2)	C6—N2	1.334 (3)
C1—C7	1.508 (3)	C6—H6	0.9500
C1—S1	1.7585 (19)	C7—C8	1.524 (3)
C2—C4	1.383 (3)	C7—C9	1.530 (3)
C2—C3	1.411 (2)	C7—H7	1.0000
C2—S1	1.7325 (19)	C8—H8A	0.9800
C3—N2	1.342 (2)	C8—H8B	0.9800
C3—N1	1.380 (2)	C8—H8C	0.9800
C4—C5	1.388 (3)	C9—H9A	0.9800
C4—H4	0.9500	C9—H9B	0.9800
C5—C6	1.400 (3)	C9—H9C	0.9800
C5—H5	0.9500		
N1—C1—C7	122.91 (17)	C8—C7—C9	111.1 (2)
N1—C1—S1	115.73 (15)	C1—C7—H7	107.6
C7—C1—S1	121.36 (14)	C8—C7—H7	107.6
C4—C2—C3	120.08 (17)	C9—C7—H7	107.6
C4—C2—S1	130.92 (14)	C7—C8—H8A	109.5
C3—C2—S1	108.98 (14)	C7—C8—H8B	109.5
N2—C3—N1	121.16 (16)	H8A—C8—H8B	109.5
N2—C3—C2	123.53 (18)	C7—C8—H8C	109.5
N1—C3—C2	115.30 (16)	H8A—C8—H8C	109.5
C2—C4—C5	116.51 (17)	H8B—C8—H8C	109.5
C2—C4—H4	121.7	C7—C9—H9A	109.5
C5—C4—H4	121.7	C7—C9—H9B	109.5

C4—C5—C6	119.6 (2)	H9A—C9—H9B	109.5
C4—C5—H5	120.2	C7—C9—H9C	109.5
C6—C5—H5	120.2	H9A—C9—H9C	109.5
N2—C6—C5	124.73 (18)	H9B—C9—H9C	109.5
N2—C6—H6	117.6	C1—N1—C3	110.99 (16)
C5—C6—H6	117.6	C6—N2—C3	115.54 (16)
C1—C7—C8	112.90 (18)	C2—S1—C1	89.00 (9)
C1—C7—C9	109.85 (15)		

C4—C2—C3—N2	−0.3 (3)	C7—C1—N1—C3	−179.88 (16)
S1—C2—C3—N2	−179.06 (14)	S1—C1—N1—C3	0.6 (2)
C4—C2—C3—N1	178.46 (16)	N2—C3—N1—C1	178.62 (17)
S1—C2—C3—N1	−0.3 (2)	C2—C3—N1—C1	−0.1 (2)
C3—C2—C4—C5	0.4 (3)	C5—C6—N2—C3	0.0 (3)
S1—C2—C4—C5	178.89 (15)	N1—C3—N2—C6	−178.61 (17)
C2—C4—C5—C6	−0.4 (3)	C2—C3—N2—C6	0.1 (3)
C4—C5—C6—N2	0.2 (3)	C4—C2—S1—C1	−178.11 (19)
N1—C1—C7—C8	169.47 (19)	C3—C2—S1—C1	0.50 (14)
S1—C1—C7—C8	−11.0 (2)	N1—C1—S1—C2	−0.64 (15)
N1—C1—C7—C9	−65.9 (3)	C7—C1—S1—C2	179.79 (16)
S1—C1—C7—C9	113.64 (19)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···N1^{i}	0.95	2.51	3.391 (2)	153

Symmetry code: (i) −*x*+1/2, *y*+1/2, *z*+1/2.