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

Gamal A. El-Hiti,[a]* Keith Smith,[b] Amany S. Hegazy,[b] Saud A. Alanazi[a] and Benson M. Kariuki[b]*

[a]Cornea Research Chair, Department of Optometry, College of Applied Medical Sciences, King Saud University, PO Box 10219, Riyadh 11433, Saudi Arabia, and [b]School of Chemistry, Cardiff University, Main Building, Park Place, Cardiff CF10 3AT, Wales. *Correspondence e-mail: gelhiti@ksu.edu.sa', kariukib@cardiff.ac.uk

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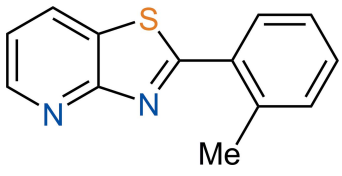
In the title molecule, $C_{13}H_{10}N_2S$, the dihedral angle between the planes through the non-H atoms of the methylbenzene and thiazolopyridine groups is 36.61 (5)°. In the crystal, the thiazolopyridine groups of inversion-related molecules overlap, with a minimum ring-centroid separation of 3.6721 (9) Å. Furthermore, the methylbenzene groups from neighbouring molecules interact edge-to-face at an angle of 71.66 (5)°. In addition, weak C—H···N hydrogen bonds form chains exending along [100].

Keywords: crystal structure; thiazolopyridine; hydrogen bonding.

CCDC reference: 1410117

1. Related literature

Various thiazolopyridine derivatives have been synthesised using different synthetic methods, see: Luo *et al.* (2015); Chaban *et al.* (2013); Leysen *et al.* (1984); Lee *et al.* (2010); Rao *et al.* (2009); Johnson *et al.* (2006); El-Hiti (2003); Smith *et al.* (1994, 1995). For the X-ray crystal structures of related compounds, see: El-Hiti *et al.* (2014; 2015); Yu *et al.* (2007).



2. Experimental

2.1. Crystal data

$C_{13}H_{10}N_2S$ M_r = 226.29

Orthorhombic, *Pbca*
a = 7.6702 (1) Å
b = 12.6492 (3) Å
c = 22.9821 (5) Å
V = 2229.77 (8) Å^3
Z = 8
Cu $K\alpha$ radiation
μ = 2.33 mm^{-1}
T = 293 K
0.26 × 0.17 × 0.05 mm

2.2. Data collection

Agilent SuperNova Dual Source diffractometer with an Atlas CCD detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2014)
T_{min} = 0.960, T_{max} = 0.989
7263 measured reflections
2234 independent reflections
1959 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.106
S = 1.03
2234 reflections
146 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···N2^{i}	0.93	2.63	3.371 (2)	137

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

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).

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: ZS2340).

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

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

S1. Introduction

Various thiazolopyridine derivatives have been synthesised using different synthetic methods (Luo *et al.*, 2015; Chaban *et al.*, 2013; Leysen *et al.*, 1984; Lee *et al.*, 2010; Rao *et al.*, 2009; Johnson *et al.*, 2006; El-Hiti, 2003; Smith *et al.*, 1994, 1995). We have synthesized 2-(2-methylphenyl)-1,3-thiazolo[4,5-*b*]pyridine in high yield (El-Hiti, 2003; Smith *et al.*, 1995) as a continuation of our research directed towards the development of novel synthetic routes towards heterocyclic derivatives. The X-ray structures for related compounds have been reported previously (El-Hiti *et al.*, 2014, 2015; Yu *et al.*, 2007).

S2. Experimental

S2.1. Synthesis and crystallization

2-(2-Methylphenyl)-1,3-thiazolo[4,5-*b*]pyridine was obtained in 89% yield from acid hydrolysis of 3-(diisopropylamino-thiocarbonylthio)-2-(2-methylbenzoylamino)pyridine under reflux (Smith *et al.*, 1995) or in 61% yield from the reaction of 3-(diisopropylaminothiocarbonylthio)-2-aminopyridine with 2-methylbenzoic acid in the presence of phosphorus oxychloride under reflux (El-Hiti, 2003). Crystallization from diethyl ether gave colourless crystals of the title compound. The NMR and mass spectral data for this compound were consistent with those reported (Smith *et al.*, 1995).

S2.2. Refinement

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.

S3. Comment

The asymmetric unit consists of one molecule of $C_{13}H_{10}N_2S$ (Fig. 1). In the molecule, the angle between the least squares planes through the nonhydrogen atoms of the methylphenyl and thiazolopyridine groups is 36.61 (5)°. In the crystal (Fig 2), the thiazolopyridine groups of adjacent inversion-related molecules are parallel and overlap fully with a minimum ring centroid separation of 3.6721 (9) Å between the 5-membered and 6-membered components of the groups (related by -*x*, -*y* +1.-*z* +1) . Methylphenyl groups from neighbouring molecules interact in an edge-to-face fashion with a dihedral angle between the rings of 71.66 (5)°. A weak intermolecular C4—H···N2^{i} contact (Table 1) forms chains of molecules extending along [100].

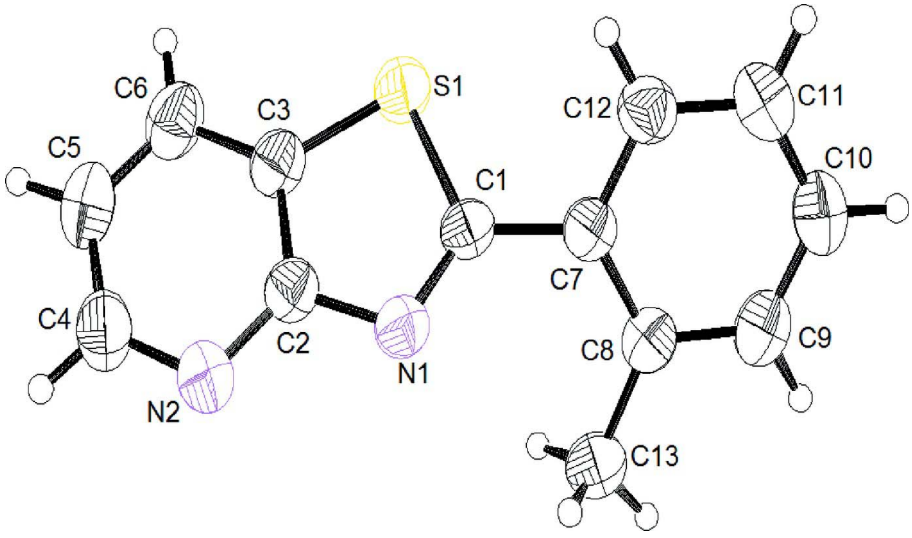

Figure 1

The asymmetric unit of $C_{13}H_{10}N_2O$ with atom labels and 50% probability displacement ellipsoids for non-hydrogen atoms.

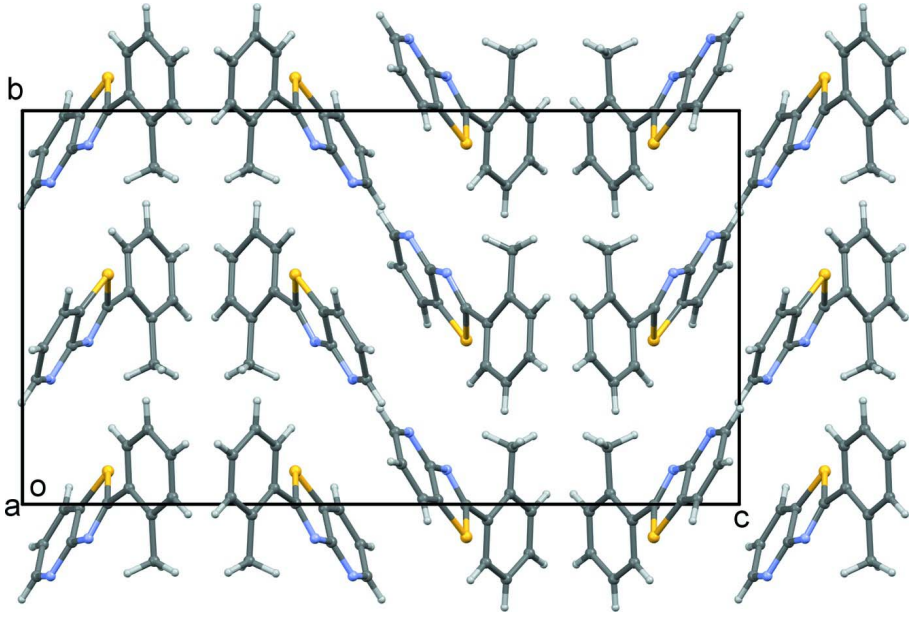

Figure 2

The crystal packing viewed along the *a* axis of the unit cell.

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

Crystal data

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$M_r = 226.29$
Orthorhombic, *Pbca*
$a = 7.6702$ (1) Å
$b = 12.6492$ (3) Å
$c = 22.9821$ (5) Å
$V = 2229.77$ (8) Å^3
$Z = 8$
$F(000) = 944$
$D_x = 1.348$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 3613 reflections

θ = 3.8–74.0°
μ = 2.33 mm^{-1}
T = 293 K

Block, colourless
0.26 × 0.17 × 0.05 mm

Data collection

Agilent SuperNova Dual Source diffractometer with an Atlas CCD detector
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2014)
T_{min} = 0.960, T_{max} = 0.989
7263 measured reflections

2234 independent reflections
1959 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 74.0°, θ_{min} = 3.9°
h = −9→6
k = −12→15
l = −28→27

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.106
S = 1.03
2234 reflections
146 parameters
0 restraints

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0633P)^2 + 0.2883P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 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	0.21789 (18)	0.50224 (11)	0.38130 (6)	0.0457 (3)
C2	0.03446 (19)	0.40362 (11)	0.43105 (6)	0.0496 (3)
C3	−0.07616 (19)	0.48902 (12)	0.42082 (6)	0.0527 (3)
C4	−0.1768 (3)	0.31679 (15)	0.48019 (8)	0.0706 (5)
H4	−0.2140	0.2579	0.5010	0.085*
C5	−0.2954 (2)	0.39752 (16)	0.47141 (8)	0.0705 (5)
H5	−0.4082	0.3917	0.4859	0.085*
C6	−0.2461 (2)	0.48645 (16)	0.44130 (8)	0.0670 (4)
H6	−0.3229	0.5422	0.4350	0.080*
C7	0.37539 (18)	0.54080 (11)	0.35116 (6)	0.0470 (3)
C8	0.48582 (19)	0.47301 (13)	0.31980 (6)	0.0527 (3)
C9	0.6265 (2)	0.51824 (15)	0.29070 (7)	0.0644 (4)
H9	0.6997	0.4749	0.2690	0.077*
C10	0.6608 (2)	0.62498 (15)	0.29292 (8)	0.0675 (4)
H10	0.7555	0.6528	0.2728	0.081*
C11	0.5547 (2)	0.69041 (14)	0.32501 (8)	0.0664 (4)
H11	0.5787	0.7623	0.3274	0.080*
C12	0.4124 (2)	0.64853 (12)	0.35362 (7)	0.0563 (4)
H12	0.3399	0.6930	0.3749	0.068*

C13	0.4583 (3)	0.35571 (14)	0.31637 (9)	0.0730 (5)
H13A	0.5337	0.3264	0.2872	0.109*
H13B	0.3391	0.3414	0.3064	0.109*
H13C	0.4845	0.3243	0.3534	0.109*
N1	0.20074 (17)	0.41287 (9)	0.40814 (5)	0.0514 (3)
N2	−0.0128 (2)	0.31754 (12)	0.46089 (7)	0.0659 (4)
S1	0.03278 (5)	0.58334 (3)	0.38069 (2)	0.06317 (17)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0444 (7)	0.0476 (7)	0.0451 (7)	0.0040 (5)	−0.0043 (5)	−0.0027 (5)
C2	0.0486 (8)	0.0529 (8)	0.0471 (7)	0.0024 (6)	0.0008 (6)	−0.0006 (6)
C3	0.0452 (7)	0.0625 (8)	0.0502 (7)	0.0045 (6)	−0.0035 (6)	0.0007 (6)
C4	0.0698 (10)	0.0746 (11)	0.0673 (10)	−0.0082 (8)	0.0169 (8)	0.0047 (8)
C5	0.0526 (9)	0.0951 (13)	0.0637 (9)	−0.0068 (8)	0.0099 (7)	−0.0010 (9)
C6	0.0474 (8)	0.0870 (12)	0.0667 (9)	0.0106 (8)	0.0017 (7)	0.0036 (8)
C7	0.0441 (7)	0.0502 (7)	0.0468 (7)	0.0005 (6)	−0.0055 (5)	0.0007 (5)
C8	0.0497 (7)	0.0567 (8)	0.0517 (8)	0.0026 (6)	−0.0004 (6)	−0.0013 (6)
C9	0.0562 (9)	0.0778 (11)	0.0591 (9)	0.0018 (8)	0.0097 (7)	−0.0019 (8)
C10	0.0600 (9)	0.0795 (11)	0.0630 (9)	−0.0143 (8)	0.0057 (7)	0.0089 (8)
C11	0.0685 (10)	0.0609 (9)	0.0700 (10)	−0.0140 (8)	−0.0009 (8)	0.0052 (8)
C12	0.0555 (8)	0.0530 (8)	0.0605 (8)	−0.0010 (7)	−0.0038 (7)	−0.0009 (6)
C13	0.0769 (12)	0.0564 (9)	0.0856 (12)	0.0054 (8)	0.0211 (9)	−0.0104 (9)
N1	0.0490 (7)	0.0508 (7)	0.0544 (7)	0.0062 (5)	0.0033 (5)	0.0039 (5)
N2	0.0665 (8)	0.0622 (8)	0.0691 (8)	0.0026 (6)	0.0133 (7)	0.0116 (7)
S1	0.0491 (3)	0.0616 (3)	0.0789 (3)	0.01227 (16)	0.00281 (17)	0.01779 (18)

Geometric parameters (Å, º)

C1—N1	1.2944 (18)	C7—C12	1.393 (2)
C1—C7	1.476 (2)	C7—C8	1.404 (2)
C1—S1	1.7518 (14)	C8—C9	1.392 (2)
C2—N2	1.337 (2)	C8—C13	1.501 (2)
C2—N1	1.3848 (19)	C9—C10	1.377 (3)
C2—C3	1.394 (2)	C9—H9	0.9300
C3—C6	1.386 (2)	C10—C11	1.375 (3)
C3—S1	1.7240 (16)	C10—H10	0.9300
C4—N2	1.334 (2)	C11—C12	1.380 (2)
C4—C5	1.382 (3)	C11—H11	0.9300
C4—H4	0.9300	C12—H12	0.9300
C5—C6	1.374 (3)	C13—H13A	0.9600
C5—H5	0.9300	C13—H13B	0.9600
C6—H6	0.9300	C13—H13C	0.9600
N1—C1—C7	126.56 (13)	C7—C8—C13	123.08 (14)
N1—C1—S1	115.65 (11)	C10—C9—C8	122.24 (16)
C7—C1—S1	117.79 (10)	C10—C9—H9	118.9

N2—C2—N1	120.92 (13)	C8—C9—H9	118.9
N2—C2—C3	123.54 (14)	C11—C10—C9	119.80 (15)
N1—C2—C3	115.54 (13)	C11—C10—H10	120.1
C6—C3—C2	119.80 (15)	C9—C10—H10	120.1
C6—C3—S1	130.81 (13)	C10—C11—C12	119.51 (16)
C2—C3—S1	109.37 (11)	C10—C11—H11	120.2
N2—C4—C5	124.51 (17)	C12—C11—H11	120.2
N2—C4—H4	117.7	C11—C12—C7	121.16 (16)
C5—C4—H4	117.7	C11—C12—H12	119.4
C6—C5—C4	119.85 (16)	C7—C12—H12	119.4
C6—C5—H5	120.1	C8—C13—H13A	109.5
C4—C5—H5	120.1	C8—C13—H13B	109.5
C5—C6—C3	116.67 (17)	H13A—C13—H13B	109.5
C5—C6—H6	121.7	C8—C13—H13C	109.5
C3—C6—H6	121.7	H13A—C13—H13C	109.5
C12—C7—C8	119.67 (14)	H13B—C13—H13C	109.5
C12—C7—C1	118.11 (13)	C1—N1—C2	110.40 (12)
C8—C7—C1	122.21 (13)	C4—N2—C2	115.61 (15)
C9—C8—C7	117.58 (15)	C3—S1—C1	89.04 (7)
C9—C8—C13	119.33 (14)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···N2^{i}	0.93	2.63	3.371 (2)	137

Symmetry code: (i) $x-1/2, -y+1/2, -z+1$.