data reports

CrossMark

2.2. Data collection

Agilent SuperNova (Dual, Cu at zero, Atlas) diffractometer	3395 measured reflections
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2014) $T_{min} = 0.721$, $T_{max} = 1.000$	1996 independent reflections 1951 reflections with $I > 2\sigma(I)$ $R_{int} = 0.016$

2.3. Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$	$\Delta\rho_{min} = -0.22$ e Å^{-3}
$wR(F^2) = 0.090$	Absolute structure: Flack x deter-
$S = 1.12$	mined using 791 quotients [(I+)-
1996 reflections	(I-)]/[(I+)+(I-)] (Parsons *et al.*,
121 parameters	2013).
H-atom parameters constrained	Absolute structure parameter:
$\Delta\rho_{max} = 0.16$ e Å^{-3}	0.027 (7)

Crystal structure of 2-*tert*-butyl-1,3-thiazolo[4,5-*b*]pyridine

Gamal A. El-Hiti,[a]* Keith Smith,[b] Amany S. Hegazy,[b] Ali M. Masmali[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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C4—H4···N1^i	0.93	2.81	3.564 (3)	138
C6—H6···N1^{ii}	0.93	2.72	3.620 (3)	164

Symmetry codes: (i) $-x+\frac{3}{2}, -y+1, z+\frac{1}{2}$; (ii) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$.

The title compound, C$_{10}$H$_{12}$N$_2$S, does not contain any strong hydrogen-bond donors but two long C—H···N contacts are observed in the crystal structure, with the most linear interaction linking molecules along [010]. The ellipsoids of the *tert*-butyl group indicate large librational motion.

Keywords: crystal structure; C—H···N contacts; 1,3-thiazolo[4,5-*b*]pyridine.

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, 2008); molecular graphics: *CHEMDRAW ultra* (Cambridge Soft, 2001); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

Acknowledgements

1. Related literature

For the synthesis of substituted thiazolopyridines, see: Smith *et al.* (1994, 1995); El-Hiti (2003); Johnson *et al.* (2006); Rao *et al.* (2009); Sahasrabudhe *et al.* (2009); Lee *et al.* (2010); Chaban *et al.* (2013). For the crystal structure of a related compound, see: Yu *et al.* (2007).
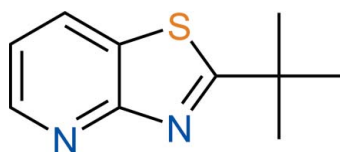
2. Experimental

2.1. Crystal data

C$_{10}$H$_{12}$N$_2$S	$V = 1030.55$ (6) Å^3
$M_r = 192.28$	$Z = 4$
Orthorhombic, $P2_12_12_1$	Cu $K\alpha$ radiation
$a = 9.4606$ (3) Å	$\mu = 2.42$ mm^{-1}
$b = 9.7999$ (3) Å	$T = 296$ K
$c = 11.1155$ (4) Å	$0.40 \times 0.29 \times 0.14$ mm

References

Agilent (2014). *CrysAlis PRO*. Agilent Technologies, Yarnton, Oxfordshire, England.
Cambridge Soft (2001). *CHEMDRAW Ultra*. Cambridge Soft Corporation, Cambridge, Massachusetts, USA.
Chaban, T. I., Ogurtsov, V. V., Chaban, I. G., Klenina, O. V. & Komarytsia, J. D. (2013). *Phosphorus Sulfur Silicon Relat. Elem.* **188**, 1611–1620.
El-Hiti, G. A. (2003). *Monatsh. Chem.* **134**, 837–841.
Johnson, S. G., Connolly, P. J. & Murray, W. V. (2006). *Tetrahedron Lett.* **47**, 4853–4856.
Lee, T., Lee, D., Lee, I. Y. & Gong, Y.-D. (2010). *J. Combin. Chem.* **12**, 95–99.
Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* B**69**, 249–259.
Rao, A. U., Palani, A., Chen, X., Huang, Y., Aslanian, R. G., West, R. E. Jr, Williams, S. M., Wu, R.-L., Hwa, J., Sondey, C. & Lachowicz, J. (2009). *Bioorg. Med. Chem. Lett.* **19**, 6176–6180.
Sahasrabudhe, K. P., Estiarte, M. A., Tan, D., Zipfel, S., Cox, M., O'Mahony, D. J. R., Edwards, W. T. & Duncton, M. A. J. (2009). *J. Heterocycl. Chem.* **46**, 1125–1131.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Smith, K., Anderson, D. & Matthews, I. (1995). *Sulfur Lett.* **18**, 79–95.
Smith, K., Lindsay, C. M., Morris, I. K., Matthews, I. & Pritchard, G. J. (1994). *Sulfur Lett.* **17**, 197–216.
Yu, Y.-Q., Wang, Y., Ni, P.-Z. & Lu, T. (2007). *Acta Cryst.* E**63**, o968–o969.

supporting information

Crystal structure of 2-*tert*-butyl-1,3-thiazolo[4,5-*b*]pyridine

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

S1. Structural commentary

Various methods have been reported for the synthesis of substituted thiazolopyridines (Smith *et al.*, 1994, 1995; El-Hiti, 2003; Johnson *et al.*, 2006; Rao *et al.*, 2009; Sahasrabudhe *et al.*, 2009; Lee *et al.*, 2010; Chaban *et al.*, 2013). In a continuation of our research focused on new synthetic routes towards substituted heterocycles we have synthesized the title compound 2-*tert*-butylthiazolo[4,5-*b*]pyridine in high yield (Smith *et al.*, 1995; El-Hiti, 2003) and now report its X-ray crystal structure. The X-ray structure for a related compound has been reported previously (Yu *et al.*, 2007). In the title compound (Fig. 1), the ellipsoids of the methyl groups of the *tert*-butyl group are large which is consistent with librational motion of the group. Assumption of a disordered model did not show significant improvement in the refinement. The molecule does not contain strong hydrogen bond donors. In the crystal, two long C—H···N contacts are observed, the most linear of which links the molecules in chains along [010] (Fig. 2).

### S2. Synthesis and crystallization

2-*tert*-Butylthiazolo[4,5-*b*]pyridine was obtained in 97% yield from acid hydrolysis of 3-(diisopropylaminothiocarbonyl-thio)-2-(pivalamido)pyridine under reflux (Smith *et al.*, 1995). The compound may also be synthesized in 66% yield from reaction of 3-(diisopropylaminothiocarbonylthio)-2-aminopyridine with 2,2-dimethylpropionic acid in the presence of phosphorus oxychloride under reflux (El-Hiti, 2003). Crystallization from a mixture of ethyl acetate and diethyl ether (1:3 by volume) gave the title compound as colourless crystals. The NMR and low and high resolution mass spectra for the title compound were consistent with those previously reported (Smith *et al.*, 1995).

S3. Refinement

Crystal data, data collection and structure refinement details are summarized in the crystal data table. The hydrogen atoms were positioned geometrically and refined using a riding model with $U_{iso}(H) = 1.2$ times U_{eq} for the atom to which they are bonded in the case of aromatic rings, NH and CH_2 groups and 1.5 times U_{eq} for the methyl hydrogens. Crystal data, data collection and structure refinement details are summarized in Table 1. The Flack parameter (Parsons *et al.*, 2013) was 0.027 (7) but is not of any structural relevance with this compound.
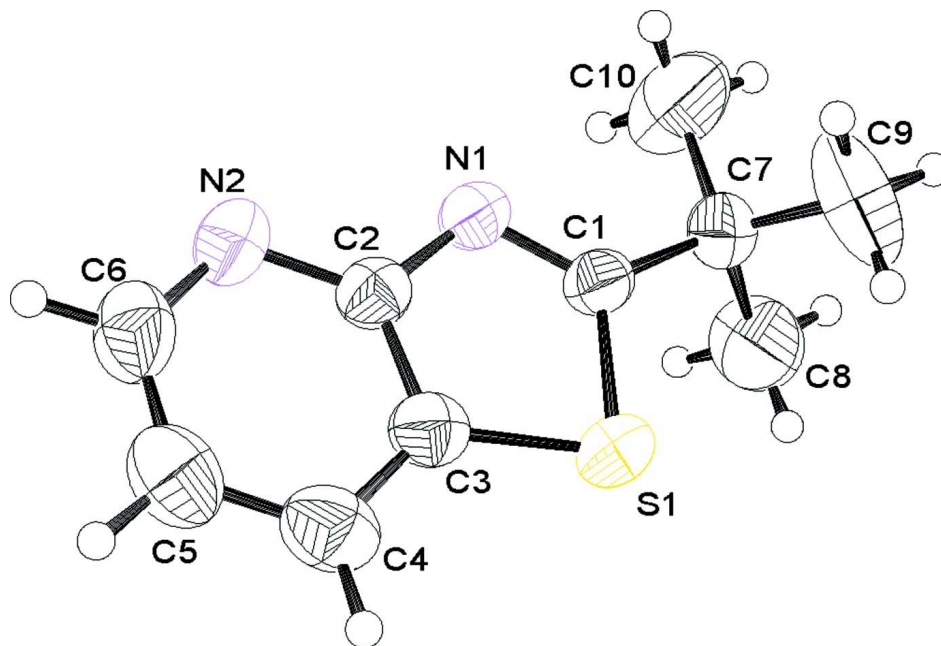
Figure 1

A molecule of the title compound showing atom labels and 50% probability displacement ellipsoids for non-H atoms.
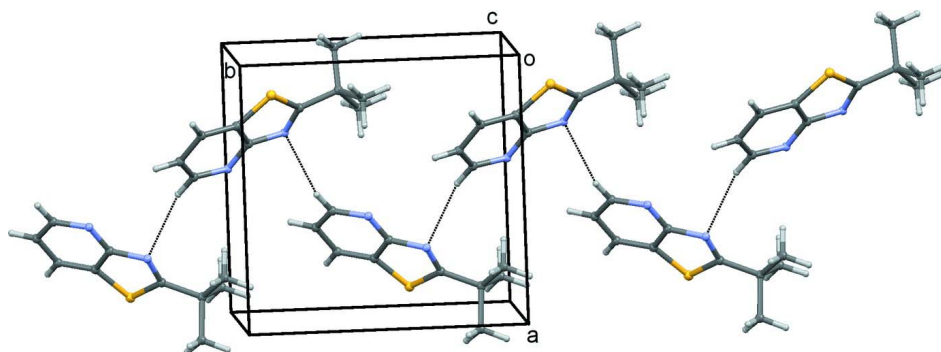
Figure 2

Crystal structure packing with the long linear C—H···N contacts shown as dashed lines.

2-*tert*-Butyl-1,3-thiazolo[4,5-*b*]pyridine

Crystal data

$C_{10}H_{12}N_2S$	$D_x = 1.239$ Mg m^{-3}
$M_r = 192.28$	Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Orthorhombic, $P2_12_12_1$	Cell parameters from 1951 reflections
$a = 9.4606$ (3) Å	$\theta = 6.0–73.4°$
$b = 9.7999$ (3) Å	$\mu = 2.42$ mm^{-1}
$c = 11.1155$ (4) Å	$T = 296$ K
$V = 1030.55$ (6) Å^3	Plate, colourless
$Z = 4$	$0.40 \times 0.29 \times 0.14$ mm
$F(000) = 408$	

Data collection

Agilent SuperNova (Dual, Cu at zero, Atlas) diffractometer	3395 measured reflections
Radiation source: SuperNova (Cu) X-ray Source	1996 independent reflections
	1951 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.016$
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2014)	$\theta_{max} = 73.4°$, $\theta_{min} = 6.0°$
	$h = -7 \rightarrow 11$
	$k = -11 \rightarrow 12$
$T_{min} = 0.721$, $T_{max} = 1.000$	$l = -13 \rightarrow 10$

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0513P)^2 + 0.0815P]$
$R[F^2 > 2\sigma(F^2)] = 0.033$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.090$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.12$	$\Delta\rho_{max} = 0.16$ e Å^{-3}
1996 reflections	$\Delta\rho_{min} = -0.22$ e Å^{-3}
121 parameters	Absolute structure: Flack x determined using
0 restraints	791 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons *et al.*, 2013).
Hydrogen site location: inferred from neighbouring sites	Absolute structure parameter: 0.027 (7)

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 (Å^2)

	x	y	z	$U_{iso}*/U_{eq}$
C1	0.8245 (2)	0.2846 (2)	0.37042 (19)	0.0458 (5)
C2	0.6967 (2)	0.4685 (2)	0.33748 (19)	0.0435 (5)
C3	0.7716 (2)	0.5154 (3)	0.4378 (2)	0.0483 (5)
C4	0.7455 (3)	0.6454 (3)	0.4822 (3)	0.0645 (6)
H4	0.7924	0.6789	0.5496	0.077*
C5	0.6470 (3)	0.7218 (3)	0.4218 (3)	0.0677 (7)
H5	0.6260	0.8097	0.4477	0.081*
C6	0.5791 (3)	0.6682 (3)	0.3225 (3)	0.0656 (7)
H6	0.5138	0.7234	0.2833	0.079*
C7	0.8831 (3)	0.1417 (2)	0.3577 (2)	0.0556 (6)
C8	0.8599 (5)	0.0641 (4)	0.4747 (3)	0.0938 (11)
H8A	0.9096	0.1090	0.5387	0.141*
H8B	0.8948	−0.0274	0.4662	0.141*
H8C	0.7608	0.0617	0.4929	0.141*
C9	1.0411 (4)	0.1506 (5)	0.3319 (5)	0.1151 (16)
H9A	1.0560	0.1983	0.2575	0.173*
H9B	1.0798	0.0603	0.3260	0.173*
H9C	1.0871	0.1990	0.3961	0.173*
C10	0.8067 (6)	0.0657 (4)	0.2577 (4)	0.124 (2)
H10A	0.7076	0.0614	0.2758	0.186*

H10B	0.8440	−0.0251	0.2513	0.186*
H10C	0.8203	0.1129	0.1829	0.186*
N1	0.72864 (19)	0.3364 (2)	0.30173 (16)	0.0453 (4)
N2	0.6004 (2)	0.5424 (2)	0.2787 (2)	0.0576 (5)
S1	0.88585 (7)	0.39028 (7)	0.48644 (5)	0.0607 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0431 (10)	0.0571 (12)	0.0371 (10)	0.0007 (10)	−0.0013 (8)	0.0016 (9)
C2	0.0423 (10)	0.0483 (11)	0.0398 (10)	−0.0043 (9)	0.0022 (8)	0.0053 (9)
C3	0.0443 (11)	0.0550 (12)	0.0457 (11)	−0.0048 (9)	0.0022 (9)	−0.0035 (9)
C4	0.0664 (15)	0.0611 (14)	0.0661 (15)	−0.0075 (11)	0.0034 (13)	−0.0160 (13)
C5	0.0707 (17)	0.0500 (13)	0.0823 (19)	−0.0008 (12)	0.0129 (15)	−0.0023 (13)
C6	0.0686 (16)	0.0542 (13)	0.0740 (17)	0.0102 (12)	0.0041 (14)	0.0157 (13)
C7	0.0564 (12)	0.0560 (13)	0.0544 (12)	0.0118 (11)	−0.0010 (11)	0.0012 (10)
C8	0.122 (3)	0.078 (2)	0.081 (2)	0.0248 (19)	0.009 (2)	0.0219 (17)
C9	0.074 (2)	0.097 (3)	0.174 (5)	0.0241 (19)	0.044 (3)	−0.007 (3)
C10	0.187 (5)	0.073 (2)	0.112 (3)	0.052 (3)	−0.072 (3)	−0.034 (2)
N1	0.0468 (9)	0.0492 (9)	0.0401 (9)	0.0001 (8)	−0.0045 (8)	0.0005 (8)
N2	0.0588 (11)	0.0570 (11)	0.0570 (12)	0.0070 (10)	−0.0078 (10)	0.0101 (9)
S1	0.0568 (3)	0.0761 (4)	0.0493 (3)	0.0096 (3)	−0.0161 (3)	−0.0112 (3)

Geometric parameters (Å, º)

C1—N1	1.290 (3)	C6—H6	0.9300
C1—C7	1.512 (3)	C7—C10	1.521 (4)
C1—S1	1.753 (2)	C7—C8	1.522 (4)
C2—N2	1.335 (3)	C7—C9	1.524 (4)
C2—N1	1.387 (3)	C8—H8A	0.9600
C2—C3	1.399 (3)	C8—H8B	0.9600
C3—C4	1.389 (4)	C8—H8C	0.9600
C3—S1	1.722 (3)	C9—H9A	0.9600
C4—C5	1.371 (4)	C9—H9B	0.9600
C4—H4	0.9300	C9—H9C	0.9600
C5—C6	1.380 (4)	C10—H10A	0.9600
C5—H5	0.9300	C10—H10B	0.9600
C6—N2	1.341 (4)	C10—H10C	0.9600
N1—C1—C7	124.6 (2)	C8—C7—C9	109.3 (3)
N1—C1—S1	115.80 (18)	C7—C8—H8A	109.5
C7—C1—S1	119.64 (17)	C7—C8—H8B	109.5
N2—C2—N1	121.0 (2)	H8A—C8—H8B	109.5
N2—C2—C3	123.9 (2)	C7—C8—H8C	109.5
N1—C2—C3	115.1 (2)	H8A—C8—H8C	109.5
C4—C3—C2	119.7 (2)	H8B—C8—H8C	109.5
C4—C3—S1	130.8 (2)	C7—C9—H9A	109.5
C2—C3—S1	109.55 (18)	C7—C9—H9B	109.5

C5—C4—C3	116.6 (3)	H9A—C9—H9B	109.5
C5—C4—H4	121.7	C7—C9—H9C	109.5
C3—C4—H4	121.7	H9A—C9—H9C	109.5
C4—C5—C6	120.0 (3)	H9B—C9—H9C	109.5
C4—C5—H5	120.0	C7—C10—H10A	109.5
C6—C5—H5	120.0	C7—C10—H10B	109.5
N2—C6—C5	124.8 (3)	H10A—C10—H10B	109.5
N2—C6—H6	117.6	C7—C10—H10C	109.5
C5—C6—H6	117.6	H10A—C10—H10C	109.5
C1—C7—C10	110.3 (2)	H10B—C10—H10C	109.5
C1—C7—C8	109.3 (2)	C1—N1—C2	110.6 (2)
C10—C7—C8	108.1 (3)	C2—N2—C6	115.0 (2)
C1—C7—C9	108.9 (3)	C3—S1—C1	88.96 (11)
C10—C7—C9	110.9 (3)		

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···N1[i]	0.93	2.81	3.564 (3)	138
C6—H6···N1[ii]	0.93	2.72	3.620 (3)	164

Symmetry codes: (i) −*x*+3/2, −*y*+1, *z*+1/2; (ii) −*x*+1, *y*+1/2, −*z*+1/2.