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

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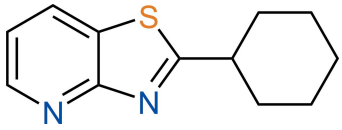
In the title compound, $C_{12}H_{14}N_2S$, the cyclohexane ring adopts a chair conformation with the exocyclic C—C bond in an equatorial orientation. The mean plane through the cyclohexane ring (all atoms) is twisted from the thiazolopyridine ring system (r.m.s. deviation = 0.013 Å) by 39.57 (6)°. In the crystal, molecules form (100) sheets, although there are no specific directional interactions between them. The crystal stucture was refined as a two-component perfect twin.

Keywords: crystal structure; cyclohexane; thiazolopyridine derivatives; thiazolo[4,5-*b*]pyridine.

CCDC reference: 1430576

1. Related literature

For background to the uses of thiazolopyridine derivatives, see: Leysen *et al.* (1984). For a related structure reported by us and further references, see: El-Hiti *et al.* (2015). For the first report of this compound and spectroscopic data, see: Smith *et al.* (1995).



2. Experimental

2.1. Crystal data

$C_{12}H_{14}N_2S$
M_r = 218.31
Monoclinic, $P2_1/n$
a = 7.8884 (5) Å
b = 11.8079 (7) Å
c = 12.2134 (6) Å
β = 100.589 (6)°
V = 1118.25 (11) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 2.29 mm^{-1}
T = 293 K
0.27 × 0.17 × 0.14 mm

2.2. Data collection

Agilent SuperNova Dual Source diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2014)
T_{min} = 0.592, T_{max} = 1.000
7328 measured reflections
3913 independent reflections
3429 reflections with $I > 2\sigma(I)$
R_{int} = 0.015

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.180
S = 1.03
3913 reflections
136 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Data collection: *CrysAlis PRO* (Agilent, 2014); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (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: HB7518).

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supporting information

Acta Cryst. (2015). E**71**, o866 [doi:10.1107/S2056989015019106]

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

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S1. Chemical context

Thiazolopyridine derivatives have been reported to exhibit interesting biological activities (Leysen *et al.*, 1984). As part of our ongoing studies in this area (El-Hiti *et al.*, 2015), we now report the structure of the title compound.

S2. Structural commentary

The asymmetric unit comprises one molecule of $C_{12}H_{14}N_2S$ (Fig. 1). The cyclohexane ring is in the chair conformation in the molecule. The least squares plane through the cyclohexane ring is twisted from the thiazolopyridine group by 39.57 (6)°. In the crystal structure, the molecular axes are aligned along [001] (Fig. 2).

S3. Synthesis and crystallization

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2-Cyclohexyl-1,3-thiazolo[4,5-*b*]pyridine was obtained in quantitative yield from acid hydrolysis (HCl, 5 M) of 3-(diisopropylaminothiocarbonylthio)-2-(cyclohexylcarbonylamino)pyridine under reflux for 5 h (Smith *et al.*, 1995). The compound may also be synthesized in 94% yield from acid hydrolysis (5 M HCl, 5 h reflux) of 3-(diisopropylaminothiocarbonylthio)-2-(*bis*(cyclohexylcarbony)lamino) pyridine (Smith *et al.*, 1995). Crystallization of the crude product from diethyl ether gave the title compound as colourless crystals. Spectroscopic and analytical data are consistent with those reported (Smith *et al.*, 1995).

S4. Refinement details

The data were twinned and HKLF5 in Shelxl 2013 was used. 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.

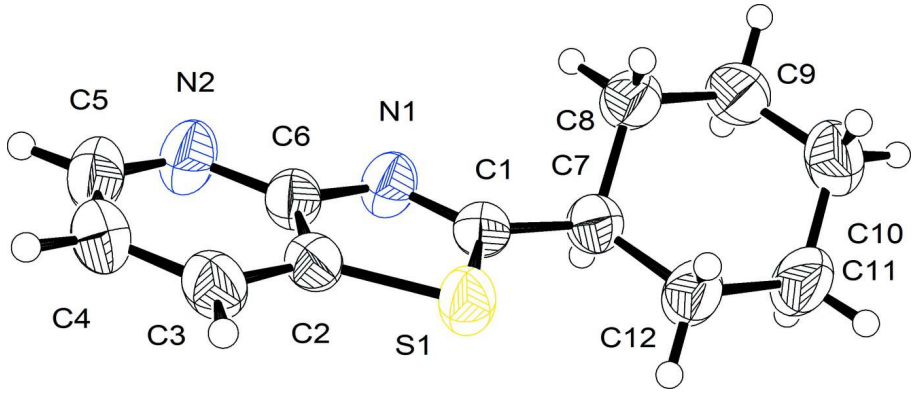

Figure 1

The asymmetric unit of $C_{12}H_{14}N_2S$ with 50% probability displacement ellipsoids for nonhydrogen atoms.

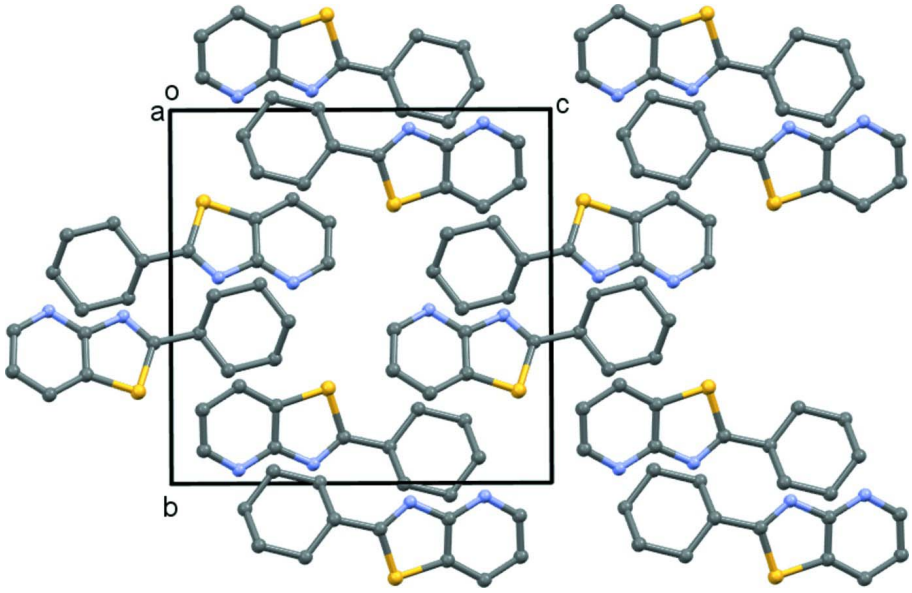

Figure 2

Crystal packing viewed along the *a* axis.

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

Crystal data

$C_{12}H_{14}N_2S$
$M_r = 218.31$
Monoclinic, $P2_1/n$
$a = 7.8884$ (5) Å
$b = 11.8079$ (7) Å
$c = 12.2134$ (6) Å
$\beta = 100.589$ (6)°
$V = 1118.25$ (11) Å^3
$Z = 4$
$F(000) = 464$
$D_x = 1.297$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 1721 reflections
$\theta = 6.2$–$73.9°$
$\mu = 2.29$ mm^{-1}
$T = 293$ K
Needle, colourless
0.27 × 0.17 × 0.14 mm

Data collection

Agilent SuperNova Dual Source diffractometer with an Atlas detector
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2014)
$T_{min} = 0.592$, $T_{max} = 1.000$
7328 measured reflections
3913 independent reflections
3429 reflections with $I > 2\sigma(I)$
$R_{int} = 0.015$
$\theta_{max} = 74.2°$, $\theta_{min} = 5.3°$
$h = -9 \rightarrow 9$
$k = -14 \rightarrow 14$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.180$
$S = 1.03$
3913 reflections
136 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1345P)^2 + 0.113P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}

Special details

Experimental. Absorption correction: CrysAlisPro, Agilent Technologies, Version 1.171.37.33 (release 27-03-2014 CrysAlis171 .NET) (compiled Mar 27 2014,17:12:48) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm. Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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.

Refinement. Refined as a 2-component perfect twin.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.2814 (3)	0.12366 (19)	0.54649 (18)	0.0467 (5)
C2	0.2459 (3)	0.20807 (19)	0.72356 (18)	0.0467 (5)
C3	0.2223 (4)	0.2609 (2)	0.8209 (2)	0.0561 (6)
H3	0.1818	0.3349	0.8212	0.067*
C4	0.2619 (4)	0.1982 (3)	0.9169 (2)	0.0634 (7)
H4	0.2461	0.2291	0.9844	0.076*
C5	0.3249 (4)	0.0897 (3)	0.9136 (2)	0.0659 (7)
H5	0.3505	0.0501	0.9805	0.079*
C6	0.3115 (3)	0.0972 (2)	0.72741 (18)	0.0472 (5)
C7	0.2823 (3)	0.0976 (2)	0.42621 (18)	0.0501 (5)
H7	0.3994	0.0741	0.4202	0.060*
C8	0.1612 (4)	−0.0018 (2)	0.3883 (2)	0.0616 (7)
H8A	0.1972	−0.0671	0.4350	0.074*
H8B	0.0450	0.0181	0.3967	0.074*
C9	0.1620 (4)	−0.0321 (2)	0.2671 (2)	0.0654 (7)
H9A	0.2758	−0.0588	0.2600	0.079*
H9B	0.0805	−0.0929	0.2443	0.079*
C10	0.1149 (4)	0.0680 (3)	0.1922 (2)	0.0706 (8)
H10A	−0.0030	0.0901	0.1937	0.085*
H10B	0.1218	0.0471	0.1163	0.085*
C11	0.2341 (5)	0.1671 (3)	0.2281 (2)	0.0856 (11)
H11A	0.1964	0.2320	0.1811	0.103*
H11B	0.3501	0.1478	0.2188	0.103*
C12	0.2352 (5)	0.1982 (2)	0.3501 (2)	0.0709 (8)
H12A	0.3176	0.2587	0.3723	0.085*
H12B	0.1220	0.2257	0.3577	0.085*
N1	0.3309 (3)	0.05196 (17)	0.62604 (16)	0.0529 (5)
N2	0.3519 (3)	0.0371 (2)	0.82184 (17)	0.0616 (6)
S1	0.20709 (8)	0.25440 (5)	0.58736 (5)	0.0545 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0509 (11)	0.0426 (11)	0.0457 (11)	−0.0033 (9)	0.0065 (9)	−0.0001 (8)
C2	0.0516 (11)	0.0423 (11)	0.0436 (11)	−0.0036 (9)	0.0021 (8)	−0.0008 (9)

C3	0.0664 (15)	0.0505 (13)	0.0496 (13)	−0.0023 (11)	0.0058 (11)	−0.0090 (10)
C4	0.0767 (17)	0.0704 (18)	0.0419 (12)	−0.0073 (13)	0.0076 (11)	−0.0079 (11)
C5	0.0794 (17)	0.0733 (18)	0.0425 (13)	0.0014 (13)	0.0047 (11)	0.0092 (11)
C6	0.0507 (12)	0.0475 (12)	0.0425 (11)	0.0013 (9)	0.0060 (8)	0.0012 (9)
C7	0.0547 (13)	0.0510 (13)	0.0453 (11)	−0.0017 (9)	0.0107 (9)	−0.0023 (9)
C8	0.0855 (18)	0.0504 (13)	0.0502 (13)	−0.0138 (13)	0.0160 (12)	−0.0044 (10)
C9	0.0846 (18)	0.0552 (15)	0.0563 (15)	−0.0118 (13)	0.0121 (12)	−0.0137 (11)
C10	0.0842 (19)	0.0764 (19)	0.0473 (13)	0.0011 (15)	0.0020 (12)	−0.0089 (13)
C11	0.145 (3)	0.0712 (19)	0.0416 (13)	−0.030 (2)	0.0189 (15)	0.0008 (12)
C12	0.116 (2)	0.0519 (15)	0.0442 (13)	−0.0148 (15)	0.0133 (13)	−0.0002 (11)
N1	0.0672 (12)	0.0472 (10)	0.0450 (10)	0.0074 (9)	0.0121 (8)	0.0036 (8)
N2	0.0795 (14)	0.0581 (13)	0.0467 (11)	0.0120 (11)	0.0104 (9)	0.0103 (9)
S1	0.0753 (5)	0.0424 (4)	0.0425 (4)	0.0053 (2)	0.0017 (3)	0.0013 (2)

Geometric parameters (Å, º)

C1—N1	1.294 (3)	C7—H7	0.9800
C1—C7	1.502 (3)	C8—C9	1.524 (4)
C1—S1	1.756 (2)	C8—H8A	0.9700
C2—C3	1.385 (3)	C8—H8B	0.9700
C2—C6	1.405 (3)	C9—C10	1.499 (4)
C2—S1	1.724 (2)	C9—H9A	0.9700
C3—C4	1.374 (4)	C9—H9B	0.9700
C3—H3	0.9300	C10—C11	1.515 (4)
C4—C5	1.378 (4)	C10—H10A	0.9700
C4—H4	0.9300	C10—H10B	0.9700
C5—N2	1.332 (3)	C11—C12	1.534 (4)
C5—H5	0.9300	C11—H11A	0.9700
C6—N2	1.342 (3)	C11—H11B	0.9700
C6—N1	1.383 (3)	C12—H12A	0.9700
C7—C12	1.512 (4)	C12—H12B	0.9700
C7—C8	1.530 (3)		
N1—C1—C7	123.0 (2)	H8A—C8—H8B	108.0
N1—C1—S1	115.61 (17)	C10—C9—C8	111.3 (2)
C7—C1—S1	121.33 (17)	C10—C9—H9A	109.4
C3—C2—C6	119.8 (2)	C8—C9—H9A	109.4
C3—C2—S1	131.07 (19)	C10—C9—H9B	109.4
C6—C2—S1	109.08 (17)	C8—C9—H9B	109.4
C4—C3—C2	116.4 (2)	H9A—C9—H9B	108.0
C4—C3—H3	121.8	C9—C10—C11	111.2 (2)
C2—C3—H3	121.8	C9—C10—H10A	109.4
C3—C4—C5	120.2 (2)	C11—C10—H10A	109.4
C3—C4—H4	119.9	C9—C10—H10B	109.4
C5—C4—H4	119.9	C11—C10—H10B	109.4
N2—C5—C4	124.9 (2)	H10A—C10—H10B	108.0
N2—C5—H5	117.6	C10—C11—C12	111.0 (3)
C4—C5—H5	117.6	C10—C11—H11A	109.4

N2—C6—N1	121.2 (2)	C12—C11—H11A	109.4
N2—C6—C2	123.3 (2)	C10—C11—H11B	109.4
N1—C6—C2	115.5 (2)	C12—C11—H11B	109.4
C1—C7—C12	113.3 (2)	H11A—C11—H11B	108.0
C1—C7—C8	109.77 (19)	C7—C12—C11	111.5 (3)
C12—C7—C8	110.3 (2)	C7—C12—H12A	109.3
C1—C7—H7	107.8	C11—C12—H12A	109.3
C12—C7—H7	107.8	C7—C12—H12B	109.3
C8—C7—H7	107.8	C11—C12—H12B	109.3
C9—C8—C7	111.1 (2)	H12A—C12—H12B	108.0
C9—C8—H8A	109.4	C1—N1—C6	110.6 (2)
C7—C8—H8A	109.4	C5—N2—C6	115.3 (2)
C9—C8—H8B	109.4	C2—S1—C1	89.19 (10)
C7—C8—H8B	109.4		