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7-Azathieno[3,2-*c*]cinnoline

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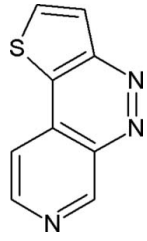
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.043; wR factor = 0.109; data-to-parameter ratio = 13.8.

The title compound, also known as pyrido[4,3-*e*]thieno[3,2-*c*]-pyridazine, $C_9H_5N_3S$, was crystallized from ethyl acetate. The molecule is planar and the N=N bond is 1.304 (3) Å compared with 1.306 (2) Å for the regio-isomer 7-azathieno[2,3-*c*]cinnoline and also in good agreement with similar compounds.

Related literature

For related literature, see: Allen *et al.* (1987); Barton *et al.* (1985); Hökelek *et al.* (1990, 1991*a*, 1991*b*); Hansen *et al.* (2007); Holt & Fiksdahl (2006); Stockmann & Fiksdahl (2007); Van der Meer (1972).



Experimental

Crystal data

$C_9H_5N_3S$
M_r = 187.22
Orthorhombic, $Pna2_1$
a = 13.0233 (13) Å
b = 15.969 (3) Å
c = 3.869 (1) Å
V = 804.6 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.35 mm^{-1}
T = 293 K
0.43 × 0.06 × 0.05 mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan (Jacobson, 1998)
T_{min} = 0.970, T_{max} = 0.990
5659 measured reflections
1623 independent reflections
1368 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.043

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.110
S = 0.99
1623 reflections
118 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Absolute structure: Flack (1983), 678 Friedel pairs
Flack parameter: 0.15 (13)

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *CrystalStructure*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SG2180).

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supplementary materials

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7-Azathieno[3,2-*c*]cinnoline

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Comment

The crystal structure of the title compound (I) was solved as part of a study of new tris-heterocyclic compounds with potential biological activity (Stockmann & Fiksdahl, 2007). Thieno[*c*]cinnolines (Barton *et al.*, 1985) have been described in the literature and the crystal structure of benzo[*c*]cinnoline (9,10-diazaphenanthrene) has been solved (Van der Meer,1972). A view of molecule (I) with the atomic numbering is presented in Fig 1. The bond lengths are within the normal range of such bonds (Allen *et al.*, 1987) and also in accordance with the regio-isomer thieno[2,3-*c*]-7-azacinnoline (Hansen *et al.*, 2007) and other benzo[*c*]cinnoline derivatives (Hökelek *et al.*, 1990, 1991*a*,1991*b*).

Experimental

Thieno[3,2-*c*]-7-azacinnoline (I) was prepared by intramolecular diazo coupling of the diazonium ion intermediate, made by $NOBF_4$ diazotization (Holt & Fiksdahl, 2006) of the 3-amino-4-(thiophen-2-yl)pyridine precursor. Single crystals were grown by crystallization from ethyl acetate (Stockmann & Fiksdahl, 2007).

Refinement

The H atoms were placed in idealized locations C—H = 0.93Å and refined as riding with $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

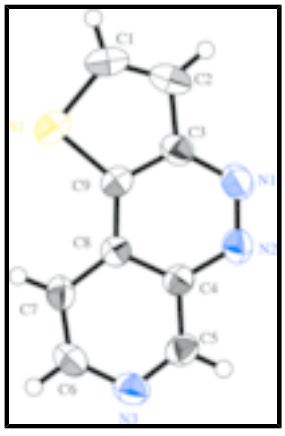

Fig. 1. A view of I with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

pyrido[4,3-*e*]thieno[3,2-*c*]pyridazine

Crystal data

$C_9H_5N_3S$ $F_{000} = 384.00$

M_r = 187.22	D_x = 1.545 Mg m^{-3}
Orthorhombic, $Pna2_1$	Mo $K\alpha$ radiation λ = 0.71070 Å
Hall symbol: P 2c -2n	Cell parameters from 2489 reflections
a = 13.0233 (13) Å	θ = 2.6–26.3º
b = 15.969 (3) Å	μ = 0.35 mm^{-1}
c = 3.869 (1) Å	T = 293 K
V = 804.6 (3) Å^3	Needle, colorless
Z = 4	0.43 × 0.06 × 0.05 mm

Data collection

Rigaku Saturn diffractometer	θ_{max} = 26.4º
ω scans	h = −16→16
Absorption correction: multi-scan (Jacobson, 1998)	k = −19→19
T_{min} = 0.970, T_{max} = 0.990	l = −4→4
5659 measured reflections	Standard reflections: ?;
1623 independent reflections	every ? reflections
1368 reflections with $F^2 > 2\sigma(F^2)$	intensity decay: ?
R_{int} = 0.043	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0638P)^2 + 0.0832P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.043	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.110	$\Delta\rho_{max}$ = 0.24 e Å^{-3}
S = 0.99	$\Delta\rho_{min}$ = −0.20 e Å^{-3}
1623 reflections	Extinction correction: none
118 parameters	Absolute structure: Flack (1983), 678 Friedel pairs
H-atom parameters constrained	Flack parameter: 0.15 (13)

Special details

Refinement. Refinement using reflections with $F^2 > 2.0$ sigma(F^2). The weighted R-factor(wR), goodness of fit (S) and R-factor (gt) are based on F, with F set to zero for negative F. The threshold expression of $F^2 > 2.0$ sigma(F^2) is used only for calculating R-factor (gt).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
S1	0.48118 (5)	0.39555 (5)	−0.2592 (2)	0.0501 (2)
N3	0.11539 (19)	0.53593 (13)	0.2017 (8)	0.0518 (6)
N2	0.18936 (17)	0.31858 (12)	0.2581 (8)	0.0480 (5)
N1	0.26250 (19)	0.26614 (14)	0.1794 (8)	0.0522 (7)

C4	0.1992 (2)	0.40207 (16)	0.1722 (7)	0.0385 (6)
C8	0.28477 (19)	0.43726 (16)	0.0052 (6)	0.0358 (6)
C3	0.3492 (2)	0.29597 (17)	0.0150 (7)	0.0433 (6)
C5	0.1168 (2)	0.45587 (17)	0.2577 (9)	0.0464 (6)
H5	0.0594	0.4321	0.3619	0.056*
C7	0.2824 (2)	0.52393 (17)	−0.0661 (8)	0.0426 (6)
H7	0.3366	0.5498	−0.1805	0.051*
C9	0.3634 (2)	0.37936 (17)	−0.0712 (7)	0.0378 (6)
C6	0.1989 (2)	0.56885 (18)	0.0363 (8)	0.0489 (7)
H6	0.1986	0.6260	−0.0095	0.059*
C2	0.4355 (2)	0.2449 (2)	−0.0781 (10)	0.0562 (8)
H2	0.4392	0.1874	−0.0409	0.067*
C1	0.5099 (2)	0.2903 (2)	−0.2255 (12)	0.0573 (8)
H1	0.5714	0.2674	−0.3025	0.069*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0385 (3)	0.0645 (4)	0.0472 (4)	0.0017 (3)	0.0040 (4)	0.0007 (4)
N3	0.0461 (13)	0.0451 (13)	0.0643 (17)	0.0070 (10)	−0.0060 (14)	−0.0018 (15)
N2	0.0452 (12)	0.0423 (12)	0.0564 (14)	−0.0076 (9)	0.0008 (15)	0.0024 (13)
N1	0.0520 (14)	0.0407 (11)	0.064 (2)	−0.0023 (11)	−0.0039 (13)	0.0056 (13)
C4	0.0340 (13)	0.0422 (14)	0.0393 (18)	−0.0011 (10)	−0.0040 (11)	−0.0002 (11)
C8	0.0337 (14)	0.0381 (14)	0.0357 (14)	−0.0019 (10)	−0.0063 (11)	−0.0010 (11)
C3	0.0437 (15)	0.0386 (14)	0.0475 (17)	0.0030 (12)	−0.0063 (13)	0.0001 (13)
C5	0.0337 (14)	0.0525 (15)	0.0530 (17)	−0.0002 (11)	0.0003 (17)	0.0012 (18)
C7	0.0398 (15)	0.0418 (15)	0.0462 (16)	−0.0066 (11)	−0.0057 (13)	0.0083 (13)
C9	0.0330 (14)	0.0474 (15)	0.0331 (15)	−0.0006 (10)	−0.0037 (12)	−0.0020 (12)
C6	0.0514 (17)	0.0364 (15)	0.059 (2)	0.0013 (13)	−0.0106 (15)	0.0005 (13)
C2	0.062 (2)	0.0455 (16)	0.0614 (18)	0.0187 (15)	−0.0095 (17)	−0.0075 (16)
C1	0.0488 (17)	0.069 (2)	0.054 (2)	0.0200 (14)	0.0006 (17)	−0.012 (2)

Geometric parameters (Å, °)

S1—C9	1.718 (2)	C8—C9	1.411 (3)
S1—C1	1.726 (3)	C3—C9	1.385 (3)
N3—C5	1.297 (3)	C3—C2	1.434 (4)
N3—C6	1.366 (4)	C7—C6	1.362 (4)
N2—N1	1.304 (3)	C2—C1	1.339 (4)
N2—C4	1.380 (3)	C5—H5	0.930
N1—C3	1.381 (3)	C7—H7	0.930
C4—C8	1.406 (3)	C6—H6	0.930
C4—C5	1.414 (3)	C2—H2	0.930
C8—C7	1.412 (3)	C1—H1	0.930
C9—S1—C1	90.87 (14)	S1—C9—C3	111.4 (2)
C5—N3—C6	116.5 (2)	C8—C9—C3	118.9 (2)
N1—N2—C4	119.8 (2)	N3—C6—C7	124.7 (2)
N2—N1—C3	118.9 (2)	C3—C2—C1	111.4 (2)

N2—C4—C8	124.8 (2)	S1—C1—C2	113.8 (2)
N2—C4—C5	117.4 (2)	N3—C5—H5	117.6
C8—C4—C5	117.8 (2)	C4—C5—H5	117.6
C4—C8—C7	117.7 (2)	C8—C7—H7	120.7
C4—C8—C9	114.2 (2)	H7—C7—C6	120.8
C7—C8—C9	128.1 (2)	N3—C6—H6	117.7
N1—C3—C9	123.4 (2)	C7—C6—H6	117.7
N1—C3—C2	124.0 (2)	C3—C2—H2	124.3
C9—C3—C2	112.5 (2)	H2—C2—C1	124.3
N3—C5—C4	124.8 (2)	S1—C1—H1	123.1
C8—C7—C6	118.5 (2)	C2—C1—H1	123.1
S1—C9—C8	129.7 (2)		
C9—S1—C1—C2	−0.5 (3)	C5—C4—C8—C9	−179.94 (19)
C1—S1—C9—C8	179.8 (2)	C4—C8—C7—C6	1.4 (4)
C1—S1—C9—C3	0.6 (2)	C4—C8—C9—S1	−177.9 (2)
C5—N3—C6—C7	−1.1 (5)	C4—C8—C9—C3	1.2 (3)
C6—N3—C5—C4	2.5 (5)	C7—C8—C9—S1	2.3 (4)
N1—N2—C4—C8	−0.9 (4)	C7—C8—C9—C3	−178.5 (2)
N1—N2—C4—C5	178.8 (3)	C9—C8—C7—C6	−178.9 (2)
C4—N2—N1—C3	0.9 (4)	N1—C3—C9—S1	177.9 (2)
N2—N1—C3—C9	0.2 (4)	N1—C3—C9—C8	−1.4 (4)
N2—N1—C3—C2	178.7 (3)	N1—C3—C2—C1	−178.2 (3)
N2—C4—C8—C7	179.6 (2)	C9—C3—C2—C1	0.4 (4)
N2—C4—C8—C9	−0.2 (3)	C2—C3—C9—S1	−0.7 (3)
N2—C4—C5—N3	178.3 (3)	C2—C3—C9—C8	−180.0 (2)
C8—C4—C5—N3	−1.9 (4)	C8—C7—C6—N3	−0.8 (4)
C5—C4—C8—C7	−0.2 (3)	C3—C2—C1—S1	0.1 (3)

Fig. 1