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2-Chloro-1-(4,5,6,7-tetrahydrothieno-[3,2-*c*]pyridin-5-yl)ethanone

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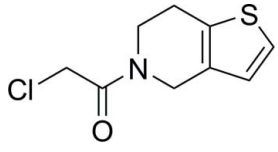
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.002 Å; R factor = 0.020; wR factor = 0.057; data-to-parameter ratio = 16.2.

In the title compound, $C_9H_{10}ClNOS$, the dihedral angle between the planar thiophene ring and 2-chloroethane moiety (r.m.s deviations of 0.003 and 0.015 Å, respectively) is 45.79 (6)°. The tetrahydropyridine ring adopts a half-chair conformation. The crystal packing reveals intermolecular C—H···O interactions.

Related literature

The title compound is an intermediate in the synthesis of thienopyridine compounds, which are characterized by anti-platelet activity. For background to thienopyridine derivatives, see: Kam & Nethery (2003). For bond-length data, see: Allen *et al.* (1987). For ring conformational analysis, see: Cremer & Pople (1975). For the preparation of 4,5,6,7-tetrahydrothieno[3,2-*c*]pyridine hydrochloride, see: Lodewijk & Khatri (1989).



Experimental

Crystal data

$C_9H_{10}ClNOS$ M_r = 215.69
Orthorhombic, $Pca2_1$
a = 10.5753 (4) Å
b = 10.8291 (4) Å
c = 8.0679 (3) Å
V = 923.94 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.59 mm^{-1}
T = 113 K
0.26 × 0.24 × 0.18 mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.861, T_{max} = 0.901
8326 measured reflections
1927 independent reflections
1850 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.057
S = 1.09
1927 reflections
119 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Absolute structure: Flack (1983), 755 Friedel pairs
Flack parameter: 0.02 (5)

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

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9A···O1^{i}	0.99	2.55	3.356 (2)	138

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

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *CrystalStructure* (Rigaku/MSC, 2005) and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KP2289).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Kam, P. C. A. & Nethery, C. M. (2003). *Anaesthesia*. **58**, 28–35.
Lodewijk, E. & Khatri, H. N. (1989). European Patent EP 0 342 118.
Rigaku/MSC (2005). *CrystalClear* and *CrystalStructure*. Rigaku/MSC Inc., The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

supplementary materials

Acta Cryst. (2010). E**66**, o3214 [**doi:10.1107/S1600536810046805**]

2-Chloro-1-(4,5,6,7-tetrahydrothieno[3,2-*c*]pyridin-5-yl)ethanone

C.-W. Yang, M. Yang, D.-K. Liu and P.-B. Wang

Comment

The thienopyridines ticlopidine and clopidogrel are inhibitors of platelet function in vivo. They are effective in preventing atherothrombotic events in cardiovascular, cerebrovascular, and peripheral vascular disease (Kam & Nethery, 2003). The crystal structure of the title compound, 2-chloro-1-(6,7-dihydrothieno [3,2-*c*]pyridin-5(4*H*)-yl)ethanone (I), an intermediate in the synthesis of some of the thienopyridines, is reported here.

In the title compound (Fig. 1) all bond lengths and angles in (I) are within normal ranges (Allen *et al.*, 1987). The thiophene ring is planar (r.m.s. deviation 0.003 Å for C8/C9/N1/O1/Cl1). The half chair conformation of the tetrahydropyridine ring is defined by the puckering parameter of φ_2=217.5 (2) ° and QT=0.5052 (15) Å (Cremer & Pople, 1975). The packing is realised by intermolecular C—H···O (Table 1) interactions.

Experimental

The synthesis of 4,5,6,7-tetrahydro-thieno[3,2-*c*]pyridine hydrochloride was reported by Lodewijk & Khatri (1989). In our experiment, 4,5,6,7-tetrahydro-thieno[3,2-*c*]pyridine was released from 4,5,6,7-tetrahydrothieno[3,2,c]pyridine hydrochloride (5.0 g, 29 mmol) by reaction with $NaHCO_3$ (7.3 g, 87 mmol) in the presence of CH_2Cl_2 (50 mL) and water (15 mL), stirred for 4 h at 273 K. The organic phase was washed with water and evaporated off under reduced pressure to get yellow oil (3.9 g, 28 mmol). The oil was dissolved in CH_2Cl_2 (50 mL) and 2-chloroacetyl chloride (3.5 g, 31 mmol) was added dropwise into the mixture. The mixture was stirred at 268 K for 5 h, the organic phase was washed with water and evaporated off under reduced pressure to get yellow oil (5.7 g) as a crude product. The product was dissolved in a mixture of petroleum ether (40 mL) and acetone (10 mL) at 273 K, and white crystals were grown slowly.

Refinement

All the H atoms was located on their parent atoms with C—H=0.95Å (aromatic CH) and 0.99Å (CH2), U_{iso} = 1.2U_{eq}(C).

Figures

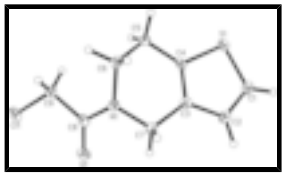

Fig. 1. The molecular structure of (I), Displacement ellipsoids are drawn at the 50% probability level.

2-Chloro-1-(4,5,6,7-tetrahydrothieno[3,2-*c*]pyridin-5-yl)ethanone

Crystal data

$C_9H_{10}ClNOS$	$F(000) = 448$
$M_r = 215.69$	$D_x = 1.551$ Mg m^{-3}
Orthorhombic, $Pca2_1$	Mo $K\alpha$ radiation, $\lambda = 0.71070$ Å
Hall symbol: P 2c -2ac	Cell parameters from 3069 reflections
$a = 10.5753$ (4) Å	$\theta = 1.9$–$27.9°$
$b = 10.8291$ (4) Å	$\mu = 0.59$ mm^{-1}
$c = 8.0679$ (3) Å	$T = 113$ K
$V = 923.94$ (6) Å^3	Block, colourless
$Z = 4$	0.26 × 0.24 × 0.18 mm

Data collection

Rigaku Saturn CCD area detector diffractometer	1927 independent reflections
Radiation source: rotating anode	1850 reflections with $I > 2\sigma(I)$
confocal	$R_{int} = 0.026$
Detector resolution: 7.31 pixels mm^{-1}	$\theta_{max} = 27.9°$, $\theta_{min} = 1.9°$
ω and φ scans	$h = -13\rightarrow13$
Absorption correction: multi-scan *CrystalClear*	$k = -14\rightarrow14$
$T_{min} = 0.861$, $T_{max} = 0.901$	$l = -10\rightarrow8$
8326 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.020$	$w = 1/[\sigma^2(F_o^2) + (0.0393P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.057$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.09$	$\Delta\rho_{max} = 0.23$ e Å^{-3}
1927 reflections	$\Delta\rho_{min} = -0.20$ e Å^{-3}
119 parameters	Extinction correction: *SHELXL*, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
1 restraint	Extinction coefficient: 0.035 (5)
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 743 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: 0.02 (5)

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.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.26658 (4)	−0.10564 (3)	0.20010 (5)	0.02247 (11)
S1	0.01605 (3)	0.55872 (3)	0.34620 (6)	0.01748 (10)
O1	0.35771 (11)	0.10076 (10)	0.39637 (14)	0.0196 (3)
N1	0.17029 (11)	0.18603 (10)	0.47077 (16)	0.0144 (3)
C1	0.14078 (15)	0.62110 (13)	0.4538 (2)	0.0186 (3)
H1	0.1563	0.7072	0.4633	0.022*
C2	0.21493 (14)	0.53226 (13)	0.52392 (19)	0.0171 (3)
H2	0.2877	0.5491	0.5891	0.021*
C3	0.17003 (14)	0.41084 (12)	0.48778 (19)	0.0141 (3)
C4	0.06333 (14)	0.40982 (12)	0.39463 (19)	0.0136 (3)
C5	−0.01059 (13)	0.29663 (11)	0.3500 (3)	0.0162 (3)
H5A	0.0058	0.2734	0.2333	0.019*
H5B	−0.1023	0.3123	0.3631	0.019*
C6	0.03142 (13)	0.19294 (12)	0.4661 (2)	0.0160 (3)
H6A	−0.0015	0.2086	0.5790	0.019*
H6B	−0.0035	0.1133	0.4269	0.019*
C7	0.22995 (14)	0.29303 (12)	0.5499 (2)	0.0161 (3)
H7A	0.3215	0.2936	0.5246	0.019*
H7B	0.2198	0.2873	0.6717	0.019*
C8	0.24161 (16)	0.10101 (12)	0.3917 (2)	0.0156 (3)
C9	0.16586 (13)	0.00648 (13)	0.29136 (19)	0.0171 (3)
H9A	0.1180	0.0496	0.2032	0.021*
H9B	0.1043	−0.0352	0.3649	0.021*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0279 (2)	0.01909 (17)	0.02041 (19)	0.00927 (14)	−0.00056 (18)	−0.00199 (16)
S1	0.01631 (18)	0.01532 (16)	0.02081 (19)	0.00237 (13)	−0.00046 (18)	0.00111 (16)
O1	0.0134 (6)	0.0224 (5)	0.0229 (6)	0.0025 (4)	0.0001 (4)	0.0021 (4)
N1	0.0134 (6)	0.0131 (5)	0.0167 (6)	−0.0018 (4)	0.0003 (5)	0.0000 (5)
C1	0.0198 (8)	0.0159 (6)	0.0201 (8)	−0.0039 (5)	0.0037 (6)	−0.0013 (6)

C2	0.0161 (8)	0.0189 (7)	0.0163 (8)	−0.0033 (5)	0.0011 (6)	−0.0025 (6)
C3	0.0130 (7)	0.0162 (6)	0.0131 (7)	−0.0011 (5)	0.0024 (6)	0.0005 (5)
C4	0.0114 (7)	0.0138 (6)	0.0157 (8)	0.0014 (5)	0.0033 (5)	−0.0002 (5)
C5	0.0116 (6)	0.0170 (6)	0.0198 (7)	−0.0001 (5)	−0.0018 (6)	−0.0029 (7)
C6	0.0127 (7)	0.0140 (6)	0.0214 (8)	−0.0023 (5)	0.0043 (6)	−0.0021 (6)
C7	0.0155 (7)	0.0150 (6)	0.0179 (8)	−0.0019 (5)	−0.0036 (6)	0.0002 (6)
C8	0.0193 (8)	0.0146 (6)	0.0128 (7)	0.0020 (5)	0.0005 (6)	0.0049 (5)
C9	0.0173 (7)	0.0165 (6)	0.0175 (7)	0.0043 (5)	0.0001 (6)	−0.0016 (5)

Geometric parameters (Å, °)

Cl1—C9	1.7751 (14)	C3—C7	1.5103 (18)
S1—C1	1.7173 (17)	C4—C5	1.4977 (18)
S1—C4	1.7329 (14)	C5—C6	1.528 (2)
O1—C8	1.2283 (19)	C5—H5A	0.9900
N1—C8	1.3503 (19)	C5—H5B	0.9900
N1—C7	1.4658 (17)	C6—H6A	0.9900
N1—C6	1.4710 (18)	C6—H6B	0.9900
C1—C2	1.364 (2)	C7—H7A	0.9900
C1—H1	0.9500	C7—H7B	0.9900
C2—C3	1.4280 (19)	C8—C9	1.532 (2)
C2—H2	0.9500	C9—H9A	0.9900
C3—C4	1.356 (2)	C9—H9B	0.9900
C1—S1—C4	91.75 (7)	N1—C6—C5	110.08 (11)
C8—N1—C7	120.29 (12)	N1—C6—H6A	109.6
C8—N1—C6	125.47 (13)	C5—C6—H6A	109.6
C7—N1—C6	113.62 (11)	N1—C6—H6B	109.6
C2—C1—S1	111.95 (11)	C5—C6—H6B	109.6
C2—C1—H1	124.0	H6A—C6—H6B	108.2
S1—C1—H1	124.0	N1—C7—C3	110.02 (12)
C1—C2—C3	111.93 (13)	N1—C7—H7A	109.7
C1—C2—H2	124.0	C3—C7—H7A	109.7
C3—C2—H2	124.0	N1—C7—H7B	109.7
C4—C3—C2	113.42 (13)	C3—C7—H7B	109.7
C4—C3—C7	121.76 (12)	H7A—C7—H7B	108.2
C2—C3—C7	124.78 (13)	O1—C8—N1	123.06 (14)
C3—C4—C5	125.05 (13)	O1—C8—C9	122.51 (13)
C3—C4—S1	110.95 (10)	N1—C8—C9	114.41 (13)
C5—C4—S1	123.83 (11)	C8—C9—Cl1	111.26 (10)
C4—C5—C6	107.60 (13)	C8—C9—H9A	109.4
C4—C5—H5A	110.2	Cl1—C9—H9A	109.4
C6—C5—H5A	110.2	C8—C9—H9B	109.4
C4—C5—H5B	110.2	Cl1—C9—H9B	109.4
C6—C5—H5B	110.2	H9A—C9—H9B	108.0
H5A—C5—H5B	108.5		
C4—S1—C1—C2	−0.19 (13)	C7—N1—C6—C5	67.73 (16)
S1—C1—C2—C3	0.67 (18)	C4—C5—C6—N1	−48.83 (16)
C1—C2—C3—C4	−1.0 (2)	C8—N1—C7—C3	126.36 (14)
C1—C2—C3—C7	−178.84 (14)	C6—N1—C7—C3	−45.10 (16)

C2—C3—C4—C5	−174.63 (15)	C4—C3—C7—N1	10.1 (2)
C7—C3—C4—C5	3.3 (2)	C2—C3—C7—N1	−172.22 (13)
C2—C3—C4—S1	0.80 (17)	C7—N1—C8—O1	7.9 (2)
C7—C3—C4—S1	178.75 (12)	C6—N1—C8—O1	178.27 (14)
C1—S1—C4—C3	−0.36 (13)	C7—N1—C8—C9	−170.49 (12)
C1—S1—C4—C5	175.15 (15)	C6—N1—C8—C9	−0.1 (2)
C3—C4—C5—C6	16.2 (2)	O1—C8—C9—Cl1	4.40 (19)
S1—C4—C5—C6	−158.65 (11)	N1—C8—C9—Cl1	−177.22 (11)
C8—N1—C6—C5	−103.21 (16)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9A···O1^{i}	0.99	2.55	3.356 (2)	138

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

Fig. 1