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2-[(4-Chlorobenzylidene)amino]-4,5,6,7-tetrahydro-1-benzothiophene-3-carbonitrile

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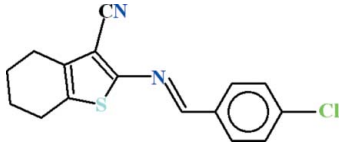
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.005 Å; R factor = 0.044; wR factor = 0.099; data-to-parameter ratio = 14.4.

In the title compound, $C_{16}H_{13}ClN_2S$, the dihedral angle between the 4-chlorobenzaldehyde moiety and the heterocyclic five-membered ring is 7.21 (17)°. In the crystal, molecules are linked by weak C—H···π interactions, generating [100] chains.

Related literature

For a related structure, see: Asiri *et al.* (2011).



Experimental

Crystal data

$C_{16}H_{13}ClN_2S$
M_r = 300.79
Orthorhombic, $P2_12_12_1$
a = 4.7815 (3) Å
b = 16.5670 (13) Å
c = 18.1658 (14) Å
V = 1439.01 (18) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.40 mm^{-1}
T = 296 K
0.35 × 0.15 × 0.12 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.931, T_{max} = 0.951
11075 measured reflections
2607 independent reflections
1821 reflections with $I > 2\sigma(I)$
R_{int} = 0.055

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.099
S = 1.02
2607 reflections
181 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Absolute structure: Flack (1983), 1053 Friedel pairs
Flack parameter: 0.03 (10)

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

Cg is the centroid of the C8–C11/S1 ring.

D—H···A	D—H	H···A	D···A	D—H···A
C13—H13A···Cg^{i}	0.97	2.99	3.841 (6)	147

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6331).

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supporting information

Acta Cryst. (2011). E**67**, o2254 [doi:10.1107/S1600536811030704]

2-[(4-Chlorobenzylidene)amino]-4,5,6,7-tetrahydro-1-benzothiophene-3-carbonitrile

Abdullah M. Asiri, Salman A. Khan and M. Nawaz Tahir

S1. Comment

We have reported the crystal structure of 2-[(benzo[1,3]dioxol-5-ylmethylene)-amino]-4,5,6,7-tetrahydro-benzo[*b*]thiophene -3-carbonitrile (Asiri *et al.*, 2011) which is related to the title compound, (I), Fig. 1.

In (I), the group A (C1–C7/CL1) of 4-chlorobenzaldehyde and the five membered ring B (C8—C11/S1) of 2-amino-4,5,6,7-tetrahydro-1-benzothiophene-3- carbonitrile group are almost planar with r. m. s. deviation of 0.0150 and 0.0110 Å, respectively. The dihedral angle between A/B is 7.21 (17)°. A C—H···π interaction (Table 1) occurs in the crystal.

S2. Experimental

A mixture of 4-chloro benzaldehyde (0.46 g, 2.4 mmol) and 2-amino-4,5,6,7-tetrahydro-benzo[*b*]thiophene-carbonitrile (0.32 g, 3.3 mmol) in ethanol (15 ml) was heated for 3 h. The progress of the reaction was monitored by TLC. The solid that separated from the cooled mixture was collected and recrystallized from a methanol-chloroform mixture (8:2) to give yellow needles of the title compound (I). Yield: 82%, m.p. 504–505 K.

S3. Refinement

The H-atoms were positioned geometrically (C–H = 0.93–0.97 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C).

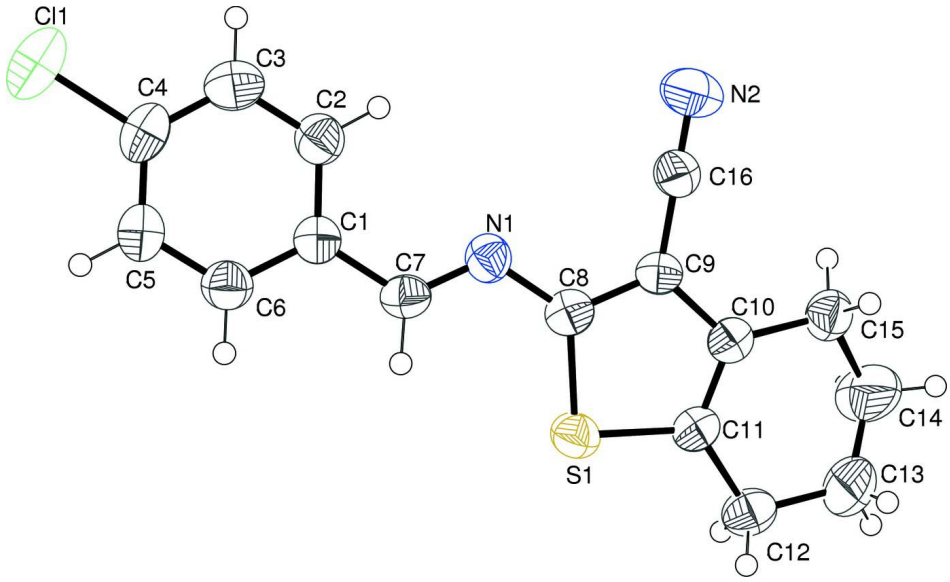

Figure 1

View of (I) showing 50% displacement ellipsoids.

2-[(4-Chlorobenzylidene)amino]-4,5,6,7-tetrahydro-1-benzothiophene- 3-carbonitrile

Crystal data

$C_{16}H_{13}ClN_2S$
$M_r = 300.79$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 4.7815\ (3)$ Å
$b = 16.5670\ (13)$ Å
$c = 18.1658\ (14)$ Å
$V = 1439.01\ (18)$ Å^3
$Z = 4$
$F(000) = 624$
$D_x = 1.388$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1821 reflections
$\theta = 3.3–25.2°$
$\mu = 0.40$ mm^{-1}
$T = 296$ K
Needle, yellow
0.35 × 0.15 × 0.12 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.20 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.931$, $T_{max} = 0.951$
11075 measured reflections
2607 independent reflections
1821 reflections with $I > 2\sigma(I)$
$R_{int} = 0.055$
$\theta_{max} = 25.2°$, $\theta_{min} = 3.3°$
$h = -5\rightarrow5$
$k = -19\rightarrow17$
$l = -21\rightarrow21$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.099$
$S = 1.02$
2607 reflections
181 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0416P)^2 + 0.1311P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}
Absolute structure: Flack (1983), 1053 Friedel pairs
Absolute structure parameter: 0.03 (10)

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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	−0.7805 (2)	−0.17259 (7)	−0.04726 (7)	0.0797 (4)
S1	0.4743 (2)	0.20961 (5)	0.08062 (4)	0.0506 (3)
N1	0.1842 (6)	0.06647 (16)	0.10865 (15)	0.0453 (10)
N2	0.4578 (9)	0.0048 (2)	0.28643 (17)	0.0787 (14)
C1	−0.1473 (7)	0.0110 (2)	0.02527 (17)	0.0406 (11)
C2	−0.2345 (8)	−0.0493 (2)	0.07260 (19)	0.0503 (12)
C3	−0.4313 (7)	−0.1048 (2)	0.0506 (2)	0.0547 (12)
C4	−0.5384 (7)	−0.1013 (2)	−0.01982 (19)	0.0513 (12)
C5	−0.4597 (8)	−0.0416 (2)	−0.06685 (19)	0.0560 (12)
C6	−0.2640 (7)	0.0135 (2)	−0.04455 (18)	0.0510 (12)
C7	0.0619 (7)	0.06949 (19)	0.04718 (19)	0.0445 (11)
C8	0.3759 (7)	0.1241 (2)	0.12951 (18)	0.0443 (12)
C9	0.5135 (8)	0.12209 (18)	0.19578 (15)	0.0393 (10)
C10	0.6876 (6)	0.19068 (19)	0.20924 (17)	0.0403 (11)
C11	0.6880 (7)	0.24227 (19)	0.15209 (17)	0.0417 (12)
C12	0.8316 (8)	0.3222 (2)	0.15026 (19)	0.0540 (12)
C13	1.0043 (12)	0.3330 (3)	0.2182 (3)	0.0947 (19)
C14	0.9144 (12)	0.2949 (3)	0.2830 (2)	0.103 (2)
C15	0.8375 (7)	0.2073 (2)	0.27953 (18)	0.0527 (12)
C16	0.4818 (9)	0.0573 (2)	0.24609 (17)	0.0484 (11)
H2	−0.15934	−0.05237	0.11974	0.0602*
H3	−0.49192	−0.14449	0.08309	0.0652*
H5	−0.53781	−0.03830	−0.11363	0.0670*
H6	−0.20794	0.05376	−0.07708	0.0609*
H7	0.10771	0.11083	0.01472	0.0534*
H12A	0.69350	0.36500	0.14716	0.0649*
H12B	0.95053	0.32546	0.10711	0.0649*
H13A	1.19183	0.31404	0.20749	0.1138*
H13B	1.01751	0.39043	0.22811	0.1138*
H14A	0.75291	0.32421	0.30118	0.1240*
H14B	1.06135	0.30081	0.31940	0.1240*
H15A	1.00517	0.17449	0.28231	0.0631*

H15B	0.71853	0.19360	0.32092	0.0631*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0610 (7)	0.0690 (7)	0.1092 (9)	−0.0156 (6)	−0.0022 (6)	−0.0261 (6)
S1	0.0612 (6)	0.0437 (5)	0.0469 (4)	−0.0018 (5)	−0.0048 (5)	0.0056 (4)
N1	0.0506 (18)	0.0409 (18)	0.0444 (16)	0.0006 (16)	−0.0036 (14)	−0.0047 (13)
N2	0.105 (3)	0.069 (2)	0.062 (2)	−0.014 (2)	−0.007 (2)	0.0196 (19)
C1	0.0409 (19)	0.036 (2)	0.045 (2)	0.0070 (17)	0.0009 (15)	−0.0040 (16)
C2	0.057 (2)	0.046 (2)	0.048 (2)	0.0009 (19)	−0.0022 (19)	−0.0042 (18)
C3	0.058 (2)	0.044 (2)	0.062 (2)	0.005 (2)	0.011 (2)	0.0012 (19)
C4	0.040 (2)	0.046 (2)	0.068 (2)	0.002 (2)	0.001 (2)	−0.0177 (19)
C5	0.054 (2)	0.060 (2)	0.054 (2)	−0.004 (2)	−0.009 (2)	−0.0066 (19)
C6	0.056 (2)	0.048 (2)	0.049 (2)	−0.001 (2)	0.001 (2)	0.0010 (18)
C7	0.050 (2)	0.038 (2)	0.0454 (18)	−0.0005 (18)	0.0050 (19)	0.0018 (16)
C8	0.046 (2)	0.040 (2)	0.047 (2)	0.0011 (18)	0.0043 (16)	−0.0030 (16)
C9	0.0423 (19)	0.0362 (18)	0.0394 (17)	0.0020 (18)	0.0040 (17)	0.0007 (14)
C10	0.0350 (18)	0.040 (2)	0.0459 (19)	0.0040 (18)	0.0049 (15)	−0.0024 (17)
C11	0.040 (2)	0.036 (2)	0.049 (2)	0.0008 (17)	0.0045 (16)	−0.0023 (17)
C12	0.056 (2)	0.042 (2)	0.064 (2)	−0.001 (2)	0.0060 (19)	0.0011 (18)
C13	0.112 (4)	0.070 (3)	0.102 (3)	−0.044 (3)	−0.038 (4)	0.007 (3)
C14	0.154 (5)	0.092 (4)	0.064 (3)	−0.063 (4)	−0.007 (3)	−0.012 (3)
C15	0.051 (2)	0.056 (2)	0.051 (2)	−0.005 (2)	−0.0019 (17)	−0.0042 (19)
C16	0.055 (2)	0.050 (2)	0.0403 (18)	−0.005 (2)	−0.0030 (19)	−0.0001 (17)

Geometric parameters (Å, º)

Cl1—C4	1.727 (4)	C11—C12	1.492 (5)
S1—C8	1.737 (3)	C12—C13	1.496 (7)
S1—C11	1.739 (3)	C13—C14	1.403 (7)
N1—C7	1.262 (4)	C14—C15	1.499 (6)
N1—C8	1.377 (4)	C2—H2	0.9300
N2—C16	1.143 (5)	C3—H3	0.9300
C1—C2	1.382 (5)	C5—H5	0.9300
C1—C6	1.386 (5)	C6—H6	0.9300
C1—C7	1.448 (5)	C7—H7	0.9300
C2—C3	1.375 (5)	C12—H12A	0.9700
C3—C4	1.379 (5)	C12—H12B	0.9700
C4—C5	1.360 (5)	C13—H13A	0.9700
C5—C6	1.369 (5)	C13—H13B	0.9700
C8—C9	1.372 (4)	C14—H14A	0.9700
C9—C10	1.430 (4)	C14—H14B	0.9700
C9—C16	1.418 (4)	C15—H15A	0.9700
C10—C11	1.345 (4)	C15—H15B	0.9700
C10—C15	1.490 (4)		
C8—S1—C11	91.79 (15)	C1—C2—H2	120.00

C7—N1—C8	121.7 (3)	C3—C2—H2	120.00
C2—C1—C6	118.0 (3)	C2—C3—H3	120.00
C2—C1—C7	121.4 (3)	C4—C3—H3	120.00
C6—C1—C7	120.6 (3)	C4—C5—H5	120.00
C1—C2—C3	120.6 (3)	C6—C5—H5	120.00
C2—C3—C4	119.7 (3)	C1—C6—H6	119.00
Cl1—C4—C3	119.2 (3)	C5—C6—H6	119.00
Cl1—C4—C5	120.1 (3)	N1—C7—H7	119.00
C3—C4—C5	120.7 (3)	C1—C7—H7	119.00
C4—C5—C6	119.2 (3)	C11—C12—H12A	110.00
C1—C6—C5	121.7 (3)	C11—C12—H12B	110.00
N1—C7—C1	122.5 (3)	C13—C12—H12A	110.00
S1—C8—N1	127.3 (2)	C13—C12—H12B	110.00
S1—C8—C9	109.8 (2)	H12A—C12—H12B	108.00
N1—C8—C9	123.0 (3)	C12—C13—H13A	108.00
C8—C9—C10	114.2 (3)	C12—C13—H13B	108.00
C8—C9—C16	122.2 (3)	C14—C13—H13A	108.00
C10—C9—C16	123.6 (3)	C14—C13—H13B	108.00
C9—C10—C11	112.0 (3)	H13A—C13—H13B	107.00
C9—C10—C15	125.0 (3)	C13—C14—H14A	108.00
C11—C10—C15	122.9 (3)	C13—C14—H14B	108.00
S1—C11—C10	112.2 (2)	C15—C14—H14A	108.00
S1—C11—C12	122.0 (2)	C15—C14—H14B	108.00
C10—C11—C12	125.6 (3)	H14A—C14—H14B	107.00
C11—C12—C13	110.0 (3)	C10—C15—H15A	110.00
C12—C13—C14	118.0 (5)	C10—C15—H15B	110.00
C13—C14—C15	118.4 (4)	C14—C15—H15A	110.00
C10—C15—C14	109.5 (3)	C14—C15—H15B	110.00
N2—C16—C9	179.5 (4)	H15A—C15—H15B	108.00

C11—S1—C8—N1	176.1 (3)	S1—C8—C9—C10	2.8 (4)
C11—S1—C8—C9	−2.1 (3)	S1—C8—C9—C16	−177.6 (3)
C8—S1—C11—C10	0.8 (3)	N1—C8—C9—C10	−175.4 (3)
C8—S1—C11—C12	−174.3 (3)	N1—C8—C9—C16	4.2 (5)
C8—N1—C7—C1	−177.9 (3)	C8—C9—C10—C11	−2.3 (4)
C7—N1—C8—S1	2.5 (5)	C8—C9—C10—C15	173.8 (3)
C7—N1—C8—C9	−179.6 (3)	C16—C9—C10—C11	178.1 (3)
C6—C1—C2—C3	−0.1 (5)	C16—C9—C10—C15	−5.8 (5)
C7—C1—C2—C3	−179.3 (3)	C9—C10—C11—S1	0.6 (4)
C2—C1—C6—C5	0.0 (5)	C9—C10—C11—C12	175.6 (3)
C7—C1—C6—C5	179.3 (3)	C15—C10—C11—S1	−175.6 (2)
C2—C1—C7—N1	3.4 (5)	C15—C10—C11—C12	−0.6 (5)
C6—C1—C7—N1	−175.9 (3)	C9—C10—C15—C14	−160.7 (3)
C1—C2—C3—C4	1.4 (5)	C11—C10—C15—C14	15.0 (5)
C2—C3—C4—Cl1	178.8 (3)	S1—C11—C12—C13	−178.9 (3)
C2—C3—C4—C5	−2.6 (5)	C10—C11—C12—C13	6.6 (5)
Cl1—C4—C5—C6	−178.9 (3)	C11—C12—C13—C14	−29.9 (6)
C3—C4—C5—C6	2.5 (5)	C12—C13—C14—C15	49.0 (7)

C4—C5—C6—C1	−1.2 (5)	C13—C14—C15—C10	−38.8 (6)

Hydrogen-bond geometry (Å, º)

Cg is the centroid of the C8–C11/S1 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C13—H13*A*···*Cg*^{i}	0.97	2.99	3.841 (6)	147

Symmetry code: (i) *x*+1, *y*, *z*.