N-Propionylthiourea

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The molecule of the title compound, $C_4H_8N_2OS$, is essentially planar; it adopts a *trans* configuration with respect to the position of the propionyl group relative to the thiono S atom about the C—N bond. The molecular structure is stabilized by an intramolecular N—H···O hydrogen bond between the propionyl O atom and the amide H atom. Molecules are linked into a two-dimensional network parallel to the $(10\bar{1})$ plane by N—H···O and N—H···S intermolecular hydrogen bonds.

Related literature

For the crystal structures of thiourea analogues, see: Yusof *et al.* (2007); Rosli *et al.* (2006). For bond-length data, see: Allen *et al.* (1987).
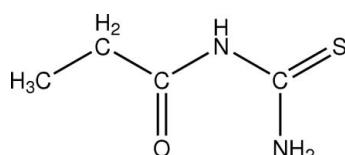
Experimental

Crystal data

$C_4H_8N_2OS$	$V = 658.8$ (3) Å^3
$M_r = 132.19$	$Z = 4$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 5.0790$ (15) Å	$\mu = 0.40$ mm^{-1}
$b = 14.342$ (4) Å	$T = 298$ (2) K
$c = 9.273$ (3) Å	$0.48 \times 0.19 \times 0.14$ mm
$\beta = 102.744$ (6)°	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	3622 measured reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)	1291 independent reflections
$T_{min} = 0.832$, $T_{max} = 0.946$	910 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.029$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$	73 parameters
$wR(F^2) = 0.119$	H-atom parameters constrained
$S = 1.02$	$\Delta\rho_{max} = 0.23$ e Å^{-3}
1291 reflections	$\Delta\rho_{min} = -0.16$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2C···O1	0.86	2.00	2.658 (3)	133
N1—H1D···O1^i	0.86	2.11	2.935 (3)	160
N2—H2D···S1^{ii}	0.86	2.57	3.409 (3)	166

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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supporting information

N-Propionylthiourea

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S1. Comment

Most carbonoylthiourea of the type, R^1HNC(S)NHR^2, such as N-butanoyl-N′- (4-nitrophenyl)thiourea (Yusof *et al.*, 2007) can be prepared from the reaction of carbonoylchloride with ammonium thiocyanate which give carbonoyl- isothio-cyanate, an intermediate for the formation of thiourea moiety when reacted with the amine compounds. However, the title compound (Fig.1) was unexpectedly obtained when the mixture of propionyl chloride and ammonium thiocyanate was stirred for 30 minutes before adding the amine compound.

  The molecule is essentially planar, with a maximum deviation of 0.021 (3) Å for atom C1 from the mean plane. The propionyl group, C1/C2/C3/O1, is *trans* relative to the thiono C4?S1 group across the C4—N1 bond. The bond lengths and angles are in normal ranges (Allen *et al.*, 1987). The molecular structure is stabilized by an intramolecular hydrogen bond, N2—H2C···O1 (Table 1), which forms a S(6) ring. In the crystal structure, the molecules are linked by N1—H1D···O^i and N2—H2D···S1^{ii} intermolecular hydrogen bonds, forming a two-dimensional network (Fig. 2) parallel to the (1 0 $\bar{1}$) plane.

S2. Experimental

A solution of ammonium thiocyanate (0.05 mol, 3.80 g) in acetone (30 ml) was added dropwise to a solution of propionyl chloride (0.05 mol, 4.63 g) in acetone (20 ml). The mixture was stirred for 30 min and the resulting light yellow solution was filtered. Single crystals of the title compound were obtained by slow evaporation of the solution (yield 90%; m.p. 420.2–421.0 K).

S3. Refinement

H atoms were positioned geometrically (C—H = 0.96 Å and N—H= 0.86 Å) and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2–1.5(methyl) U_{eq}(parent atom).
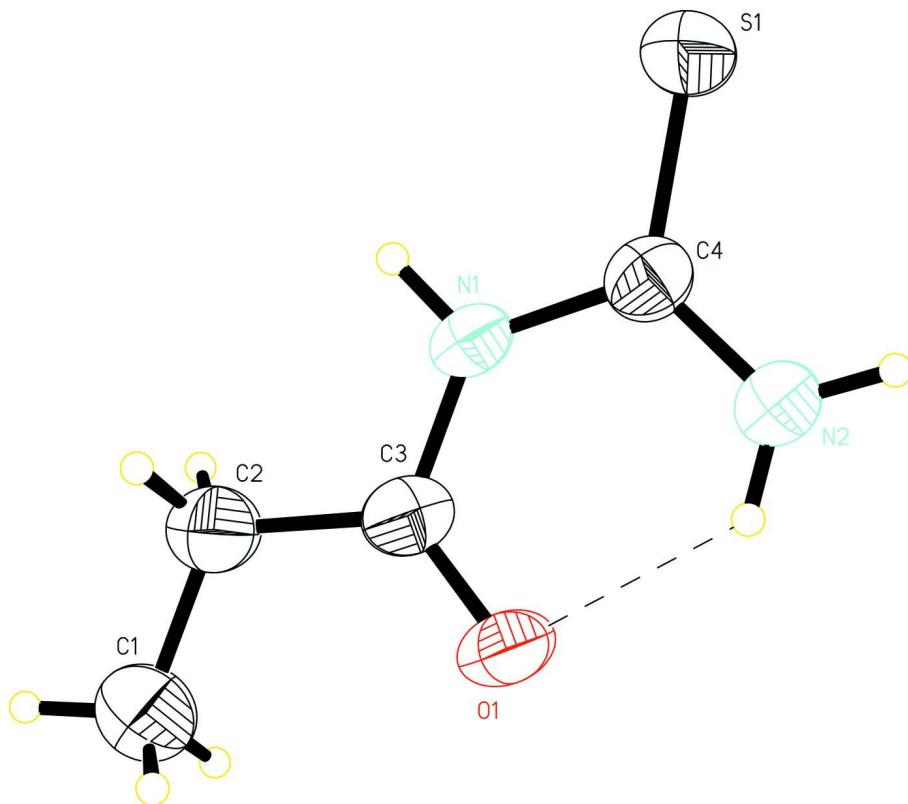
Figure 1

The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level. The dashed line indicates a hydrogen bond.
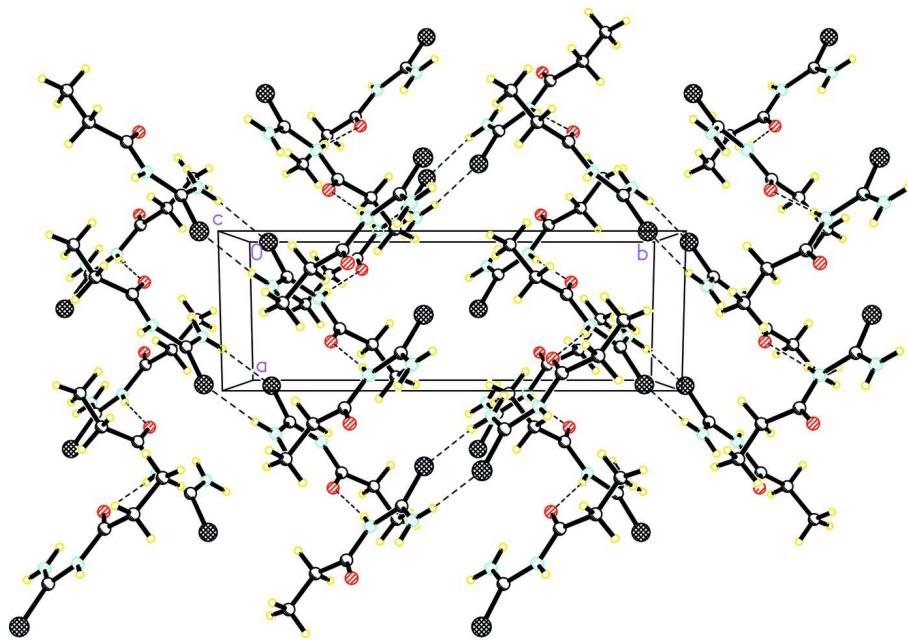
Figure 2

The molecular packing of the title compound, viewed approximately down the *c* axis. Hydrogen bonds are shown as dashed lines.

N-Propionylthiourea

Crystal data

$C_4H_8N_2OS$	$F(000) = 280$
$M_r = 132.19$	$D_x = 1.333$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 1291 reflections
$a = 5.0790$ (15) Å	$\theta = 2.6$–26.0°
$b = 14.342$ (4) Å	$\mu = 0.40$ mm^{-1}
$c = 9.273$ (3) Å	$T = 298$ K
$\beta = 102.744$ (6)°	Block, yellow
$V = 658.8$ (3) Å^3	$0.48 \times 0.19 \times 0.14$ mm
$Z = 4$	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	3622 measured reflections
	1291 independent reflections
Radiation source: fine-focus sealed tube	910 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{int} = 0.029$
Detector resolution: 83.66 pixels mm^{-1}	$\theta_{max} = 26.0°$, $\theta_{min} = 2.6°$
ω scan	$h = -6 \rightarrow 5$
Absorption correction: multi-scan	$k = -16 \rightarrow 17$
(*SADABS*; Bruker, 2000)	$l = -9 \rightarrow 11$
$T_{min} = 0.832$, $T_{max} = 0.946$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.048$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.119$	
$S = 1.02$	H-atom parameters constrained
1291 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0492P)^2 + 0.293P]$
73 parameters	where $P = (F_o^2 + 2F_c^2)/3$
0 restraints	$(\Delta/\sigma)_{max} = 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.23$ e Å^{-3}
	$\Delta\rho_{min} = -0.16$ e Å^{-3}

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}
S1	0.01989 (18)	0.06361 (6)	0.28118 (8)	0.0646 (3)
O1	0.7005 (4)	0.22101 (13)	0.58191 (18)	0.0537 (5)
N1	0.4084 (4)	0.18551 (14)	0.3657 (2)	0.0428 (6)
H1D	0.3630	0.2009	0.2739	0.051*

C4	0.2638 (5)	0.11286 (17)	0.4084 (3)	0.0413 (6)
N2	0.3300 (5)	0.08712 (15)	0.5470 (2)	0.0514 (6)
H2C	0.4587	0.1151	0.6072	0.062*
H2D	0.2448	0.0422	0.5779	0.062*
C3	0.6154 (5)	0.23647 (18)	0.4506 (3)	0.0431 (6)
C2	0.7271 (7)	0.3102 (2)	0.3675 (3)	0.0635 (9)
H2A	0.7959	0.2807	0.2892	0.076*
H2B	0.5808	0.3512	0.3212	0.076*
C1	0.9454 (7)	0.3677 (2)	0.4578 (3)	0.0706 (9)
H1A	1.0051	0.4130	0.3958	0.106*
H1B	1.0940	0.3282	0.5022	0.106*
H1C	0.8785	0.3990	0.5340	0.106*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0696 (6)	0.0701 (6)	0.0465 (5)	−0.0255 (4)	−0.0038 (4)	0.0050 (4)
O1	0.0653 (13)	0.0566 (12)	0.0324 (10)	−0.0098 (10)	−0.0040 (8)	0.0002 (8)
N1	0.0498 (14)	0.0479 (13)	0.0273 (10)	−0.0060 (11)	0.0013 (9)	0.0028 (9)
C4	0.0445 (15)	0.0408 (15)	0.0385 (14)	0.0040 (12)	0.0089 (11)	−0.0008 (11)
N2	0.0633 (16)	0.0523 (14)	0.0357 (12)	−0.0122 (12)	0.0045 (10)	0.0034 (10)
C3	0.0473 (17)	0.0420 (15)	0.0373 (14)	0.0028 (12)	0.0032 (11)	−0.0015 (11)
C2	0.073 (2)	0.063 (2)	0.0484 (17)	−0.0223 (17)	−0.0002 (15)	0.0073 (14)
C1	0.076 (2)	0.069 (2)	0.064 (2)	−0.0238 (18)	0.0095 (17)	−0.0026 (16)

Geometric parameters (Å, º)

S1—C4	1.668 (3)	C3—C2	1.492 (4)
O1—C3	1.219 (3)	C2—C1	1.483 (4)
N1—C3	1.377 (3)	C2—H2A	0.97
N1—C4	1.382 (3)	C2—H2B	0.97
N1—H1D	0.86	C1—H1A	0.96
C4—N2	1.308 (3)	C1—H1B	0.96
N2—H2C	0.86	C1—H1C	0.96
N2—H2D	0.86		
C3—N1—C4	128.6 (2)	C1—C2—C3	115.1 (2)
C3—N1—H1D	115.7	C1—C2—H2A	108.5
C4—N1—H1D	115.7	C3—C2—H2A	108.5
N2—C4—N1	117.2 (2)	C1—C2—H2B	108.5
N2—C4—S1	124.4 (2)	C3—C2—H2B	108.5
N1—C4—S1	118.37 (18)	H2A—C2—H2B	107.5
C4—N2—H2C	120.0	C2—C1—H1A	109.5
C4—N2—H2D	120.0	C2—C1—H1B	109.5
H2C—N2—H2D	120.0	H1A—C1—H1B	109.5
O1—C3—N1	122.2 (2)	C2—C1—H1C	109.5
O1—C3—C2	123.6 (2)	H1A—C1—H1C	109.5
N1—C3—C2	114.2 (2)	H1B—C1—H1C	109.5

C3—N1—C4—N2	−0.7 (4)	C4—N1—C3—C2	179.8 (3)
C3—N1—C4—S1	179.7 (2)	O1—C3—C2—C1	−2.5 (5)
C4—N1—C3—O1	0.7 (4)	N1—C3—C2—C1	178.4 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*C*···O1	0.86	2.00	2.658 (3)	133
N1—H1*D*···O1ⁱ	0.86	2.11	2.935 (3)	160
N2—H2*D*···S1ⁱⁱ	0.86	2.57	3.409 (3)	166

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