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N-Butanoyl-*N*′-(3,4-dichlorophenyl)thiourea

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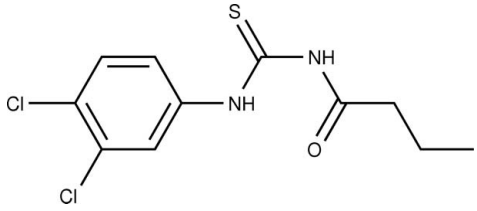
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.040; wR factor = 0.109; data-to-parameter ratio = 15.9.

The molecule in the title compound, $C_{11}H_{12}Cl_2N_2OS$, adopts a *trans–cis* configuration of the butanoyl and 3,4-dichlorophenyl groups with respect to the thiono S atom across the thiourea C—N bonds. In the crystal structure, molecules are linked into a two-dimensional network by N—H···S and C—H···O interactions.

Related literature

For related crystal structures, see: Kadir *et al.* (2007); Yusof *et al.* (2007).



Experimental

Crystal data

$C_{11}H_{12}Cl_2N_2OS$
M_r = 291.19
Triclinic, $P\overline{1}$
a = 6.0544 (12) Å
b = 10.968 (2) Å
c = 11.436 (2) Å
α = 110.404 (3)°
β = 98.028 (3)°
γ = 104.489 (3)°
V = 667.3 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.63 mm^{-1}
T = 298 (2) K
0.37 × 0.36 × 0.32 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.801, T_{max} = 0.824
6670 measured reflections
2477 independent reflections
2146 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.109
S = 1.05
2477 reflections
156 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1	0.86	1.99	2.658 (3)	133
N1—H1···S1^{i}	0.86	2.51	3.365 (2)	171
C7—H7···O1^{ii}	0.93	2.52	3.284 (3)	140
C8—H8···O1^{iii}	0.93	2.54	3.327 (3)	143

Symmetry codes: (i) $-x+1, -y+1, -z+2$; (ii) $x+1, y, z$; (iii) $-x+1, -y, -z+2$.

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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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2107).

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supplementary materials

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N-Butanoyl-*N*'-(3,4-dichlorophenyl)thiourea

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Comment

The title compound, (I), is similar to *N*-(3,4-Dichlorophenyl)-*N*'-decanoylthiourea (II), (Kadir *et al.*, 2007) and *N*-butanoyl-*N*'-(4-nitrophenyl)thiourea (III), (Yusof *et al.*, 2007) (Fig.1). The molecule also adopts *cis-trans* configuration with respect to the position of the butanoyl and 3,4-dichlorophenyl groups relative to the thiono S1 atom across their C—N bonds, respectively. The bond lengths and angles are in normal ranges and comparable to those in (II) and (III). The central thiourea (S1/N1/N2/C5) and 3,4-dichlorophenyl, (C6—C11/Cl1/Cl2) fragments are essentially planar with the maximum deviation of 0.024 (2)Å for atom N1. The dihedral angle between these fragments is 89.65 (6)°. [coplanar in (II) and 5.27 (8)° in (III)].

There is an intramolecular hydrogen bond, N2—H2···O1, closing a pseudo-six-membered ring (O1···H2—N2—C5—N1—C4—O1). In the crystal structure, the molecules are linked by intermolecular, N—H···S and C—H···O (symmetry codes as in Table 1) hydrogen bonds into two-dimensional network (Fig.2 & Table 1).

Experimental

3,4-Dichloroaniline (3.08 g, 19 mmol) in 40 ml of acetone was added dropwise to a stirred acetone solution (75 ml) of butyrylchloride (2.0 g, 19 mmol) and ammoniumthiocyanate (1.43 g, 19 mmol). The mixture was refluxed for 1 h. The resulting solution was poured into a beaker containing some ice blocks. The white precipitate was filtered off and washed with distilled water and cold ethanol before being dried under vacuum. Good quality crystals were obtained by recrystallization from DMSO.

Refinement

After their location in the difference map, all H-atoms were fixed geometrically at ideal positions and allowed to ride on the parent C or N atoms with C—H = 0.93–0.97Å and N—H = 0.86Å with U_{iso}(H)= 1.2 (CH_2 and NH) or 1.5U_{eq}(C)(CH_3).

Figures

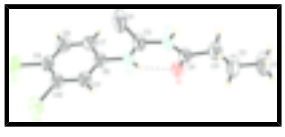

Fig. 1. : The molecular structure of (I) with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. Dashed lines indicate hydrogen bonds.

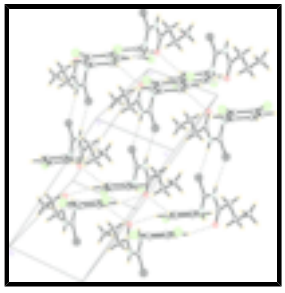

Fig. 2. : Packing diagram of compound(I), viewed down the *c* axis. The dashed lines denote the N—H···S and C—H···O hydrogen bonds.

N-Butanoyl-N'-(3,4-dichlorophenyl)thiourea

Crystal data

$C_{11}H_{12}Cl_2N_2OS$	$Z = 2$
$M_r = 291.19$	$F_{000} = 300$
Triclinic, $P\overline{1}$	$D_x = 1.449$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 6.0544$ (12) Å	Cell parameters from 883 reflections
$b = 10.968$ (2) Å	$\theta = 2.0$–25.5º
$c = 11.436$ (2) Å	$\mu = 0.63$ mm^{-1}
$\alpha = 110.404$ (3)º	$T = 298$ (2) K
$\beta = 98.028$ (3)º	Block, colourless
$\gamma = 104.489$ (3)º	0.37 × 0.36 × 0.32 mm
$V = 667.3$ (2) Å^3	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	2477 independent reflections
Radiation source: fine-focus sealed tube	2146 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.020$
Detector resolution: 83.66 pixels mm^{-1}	$\theta_{max} = 25.5$º
$T = 298(2)$ K	$\theta_{min} = 2.0$º
ω scans	$h = -7 \rightarrow 7$
Absorption correction: multi-scan (SADABS; Bruker, 2000)	$k = -13 \rightarrow 13$
$T_{min} = 0.801$, $T_{max} = 0.824$	$l = -13 \rightarrow 13$
6670 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.040$	$w = 1/[\sigma^2(F_o^2) + (0.0545P)^2 + 0.2588P]$ where $P = (F_o^2 + 2F_c^2)/3$

$wR(F^2) = 0.109$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.05$	$\Delta\rho_{max} = 0.36$ e Å^{-3}
2477 reflections	$\Delta\rho_{min} = -0.35$ e Å^{-3}
156 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997a), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.011 (3)
Secondary atom site location: difference Fourier map	

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 $F^2 > 2\text{sigma}(F^2)$ 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	0.60174 (13)	−0.26369 (8)	0.49131 (7)	0.0808 (3)
Cl2	0.17514 (12)	−0.17210 (7)	0.40676 (5)	0.0688 (2)
S1	0.56560 (16)	0.32891 (6)	0.87005 (8)	0.0889 (3)
O1	0.0254 (3)	0.16073 (14)	1.05932 (15)	0.0586 (4)
N1	0.2667 (4)	0.32607 (18)	1.01730 (18)	0.0589 (5)
H1	0.3165	0.4134	1.0389	0.071*
N2	0.2734 (3)	0.11439 (17)	0.88355 (17)	0.0554 (5)
H2	0.1664	0.0795	0.9156	0.066*
C1	−0.2070 (7)	0.4617 (3)	1.3244 (4)	0.1049 (12)
H1A	−0.1036	0.5034	1.4094	0.157*
H1B	−0.3653	0.4242	1.3290	0.157*
H1C	−0.2007	0.5296	1.2890	0.157*
C2	−0.1304 (6)	0.3479 (3)	1.2394 (3)	0.0907 (10)
H2A	−0.0668	0.3052	1.2914	0.109*
H2B	−0.2675	0.2785	1.1741	0.109*
C3	0.0481 (5)	0.3950 (2)	1.1753 (3)	0.0674 (7)
H3A	0.1909	0.4571	1.2407	0.081*
H3B	−0.0089	0.4466	1.1313	0.081*
C4	0.1083 (4)	0.2819 (2)	1.0797 (2)	0.0508 (5)
C5	0.3585 (4)	0.2502 (2)	0.9244 (2)	0.0559 (5)
C6	0.3542 (4)	0.02415 (19)	0.7878 (2)	0.0489 (5)
C7	0.5443 (4)	−0.0133 (2)	0.8248 (2)	0.0556 (5)
H7	0.6219	0.0209	0.9113	0.067*
C8	0.6189 (4)	−0.1020 (2)	0.7322 (2)	0.0586 (6)

H8	0.7476	−0.1274	0.7566	0.070*
C9	0.5047 (4)	−0.1527 (2)	0.6046 (2)	0.0518 (5)
C10	0.3152 (4)	−0.1139 (2)	0.5678 (2)	0.0483 (5)
C11	0.2388 (4)	−0.0254 (2)	0.6597 (2)	0.0499 (5)
H11	0.1107	0.0005	0.6353	0.060*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0879 (5)	0.0781 (5)	0.0821 (5)	0.0464 (4)	0.0457 (4)	0.0156 (3)
Cl2	0.0829 (4)	0.0676 (4)	0.0501 (3)	0.0267 (3)	0.0209 (3)	0.0135 (3)
S1	0.1366 (7)	0.0404 (3)	0.0956 (5)	0.0213 (4)	0.0829 (5)	0.0185 (3)
O1	0.0664 (9)	0.0400 (8)	0.0654 (9)	0.0145 (7)	0.0292 (8)	0.0133 (7)
N1	0.0828 (13)	0.0348 (9)	0.0577 (11)	0.0174 (9)	0.0369 (10)	0.0105 (8)
N2	0.0676 (11)	0.0360 (9)	0.0567 (10)	0.0130 (8)	0.0317 (9)	0.0083 (8)
C1	0.131 (3)	0.0685 (18)	0.133 (3)	0.0415 (19)	0.095 (3)	0.0305 (19)
C2	0.118 (2)	0.0557 (15)	0.115 (2)	0.0337 (15)	0.081 (2)	0.0299 (16)
C3	0.0826 (17)	0.0452 (12)	0.0745 (16)	0.0201 (11)	0.0445 (14)	0.0150 (11)
C4	0.0572 (12)	0.0414 (11)	0.0495 (11)	0.0138 (9)	0.0196 (9)	0.0123 (9)
C5	0.0755 (15)	0.0403 (11)	0.0508 (12)	0.0187 (10)	0.0285 (11)	0.0121 (9)
C6	0.0571 (12)	0.0336 (9)	0.0516 (11)	0.0118 (8)	0.0240 (9)	0.0099 (9)
C7	0.0574 (13)	0.0519 (12)	0.0512 (12)	0.0136 (10)	0.0156 (10)	0.0154 (10)
C8	0.0543 (12)	0.0569 (13)	0.0698 (15)	0.0243 (10)	0.0226 (11)	0.0243 (11)
C9	0.0570 (12)	0.0407 (10)	0.0596 (13)	0.0178 (9)	0.0294 (10)	0.0152 (9)
C10	0.0560 (12)	0.0365 (10)	0.0483 (11)	0.0112 (9)	0.0212 (9)	0.0116 (8)
C11	0.0542 (12)	0.0398 (10)	0.0571 (12)	0.0175 (9)	0.0215 (10)	0.0163 (9)

Geometric parameters (Å, °)

Cl1—C9	1.725 (2)	C2—H2A	0.9700
Cl2—C10	1.729 (2)	C2—H2B	0.9700
S1—C5	1.660 (2)	C3—C4	1.504 (3)
O1—C4	1.220 (3)	C3—H3A	0.9700
N1—C4	1.365 (3)	C3—H3B	0.9700
N1—C5	1.382 (3)	C6—C7	1.376 (3)
N1—H1	0.8600	C6—C11	1.378 (3)
N2—C5	1.329 (3)	C7—C8	1.381 (3)
N2—C6	1.433 (2)	C7—H7	0.9300
N2—H2	0.8600	C8—C9	1.370 (3)
C1—C2	1.512 (4)	C8—H8	0.9300
C1—H1A	0.9600	C9—C10	1.382 (3)
C1—H1B	0.9600	C10—C11	1.382 (3)
C1—H1C	0.9600	C11—H11	0.9300
C2—C3	1.475 (3)		
C4—N1—C5	129.10 (18)	O1—C4—N1	122.30 (19)
C4—N1—H1	115.5	O1—C4—C3	123.51 (19)
C5—N1—H1	115.5	N1—C4—C3	114.19 (18)
C5—N2—C6	122.09 (18)	N2—C5—N1	116.59 (19)

C5—N2—H2	119.0	N2—C5—S1	123.20 (16)
C6—N2—H2	119.0	N1—C5—S1	120.20 (16)
C2—C1—H1A	109.5	C7—C6—C11	120.73 (19)
C2—C1—H1B	109.5	C7—C6—N2	119.7 (2)
H1A—C1—H1B	109.5	C11—C6—N2	119.5 (2)
C2—C1—H1C	109.5	C6—C7—C8	119.4 (2)
H1A—C1—H1C	109.5	C6—C7—H7	120.3
H1B—C1—H1C	109.5	C8—C7—H7	120.3
C3—C2—C1	113.7 (2)	C9—C8—C7	120.4 (2)
C3—C2—H2A	108.8	C9—C8—H8	119.8
C1—C2—H2A	108.8	C7—C8—H8	119.8
C3—C2—H2B	108.8	C8—C9—C10	119.96 (19)
C1—C2—H2B	108.8	C8—C9—Cl1	119.31 (17)
H2A—C2—H2B	107.7	C10—C9—Cl1	120.73 (17)
C2—C3—C4	114.7 (2)	C11—C10—C9	120.1 (2)
C2—C3—H3A	108.6	C11—C10—Cl2	119.26 (17)
C4—C3—H3A	108.6	C9—C10—Cl2	120.65 (15)
C2—C3—H3B	108.6	C6—C11—C10	119.4 (2)
C4—C3—H3B	108.6	C6—C11—H11	120.3
H3A—C3—H3B	107.6	C10—C11—H11	120.3
C1—C2—C3—C4	−173.9 (3)	N2—C6—C7—C8	178.98 (19)
C5—N1—C4—O1	0.8 (4)	C6—C7—C8—C9	−0.2 (3)
C5—N1—C4—C3	−179.7 (2)	C7—C8—C9—C10	0.8 (3)
C2—C3—C4—O1	−2.9 (4)	C7—C8—C9—Cl1	−179.83 (17)
C2—C3—C4—N1	177.6 (3)	C8—C9—C10—C11	−0.9 (3)
C6—N2—C5—N1	−179.8 (2)	Cl1—C9—C10—C11	179.70 (16)
C6—N2—C5—S1	−0.3 (3)	C8—C9—C10—Cl2	177.73 (17)
C4—N1—C5—N2	4.1 (4)	Cl1—C9—C10—Cl2	−1.6 (3)
C4—N1—C5—S1	−175.5 (2)	C7—C6—C11—C10	0.2 (3)
C5—N2—C6—C7	88.9 (3)	N2—C6—C11—C10	−179.12 (18)
C5—N2—C6—C11	−91.8 (3)	C9—C10—C11—C6	0.5 (3)
C11—C6—C7—C8	−0.3 (3)	Cl2—C10—C11—C6	−178.23 (15)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O1	0.86	1.99	2.658 (3)	133
N1—H1···S1^{i}	0.86	2.51	3.365 (2)	171
C7—H7···O1^{ii}	0.93	2.52	3.284 (3)	140
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Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+2; (ii) *x*+1, *y*, *z*; (iii) −*x*+1, −*y*, −*z*+2.

Fig. 1

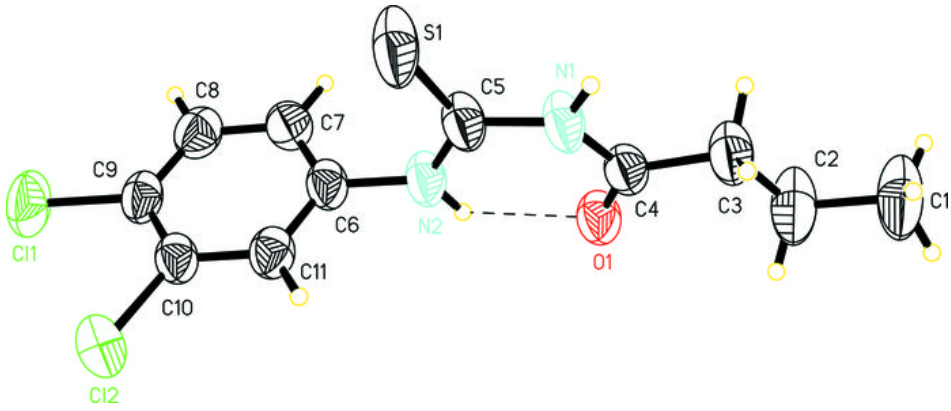

Fig. 2