Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N-Butanoyl-*N*′-(4-nitrophenyl)thiourea

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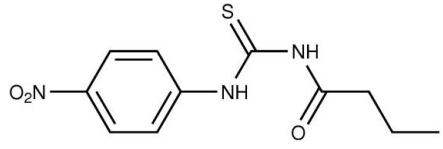
Received 18 July 2007; accepted 5 August 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.043; wR factor = 0.128; data-to-parameter ratio = 15.5.

The molecule in the title compound, $C_{11}H_{13}N_3O_3S$, adopts a *trans–cis* configuration with respect to the positions of the butanoyl and 4-nitrophenyl groups relative to the S atom across their respective C—N bonds. In the crystal structure, molecules are linked into two-dimensional networks by N—H···S and C—H···O interactions.

Related literature

For some related structures, see: Yusof *et al.* (2006, 2007*a*,*b*).



Experimental

Crystal data

$C_{11}H_{13}N_3O_3S$
M_r = 267.30
Monoclinic, $P2_1/c$
a = 4.9002 (12) Å
b = 12.385 (3) Å
c = 21.774 (5) Å
β = 100.727 (6)°
V = 1298.3 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 293 (2) K
0.51 × 0.50 × 0.46 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.882, T_{max} = 0.892
7048 measured reflections
2540 independent reflections
1933 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.128
S = 1.03
2540 reflections
164 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2*A*···O1	0.86	1.89	2.629 (2)	143
N1—H1*A*···S1^{i}	0.86	2.62	3.4579 (18)	164
C7—H7*A*···O2^{ii}	0.93	2.48	3.397 (3)	168

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

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

The authors thank the Malaysian Government, Universiti Kebangsaan Malaysia and Universiti Malaysia Terengganu for research grants IRPA Nos. 09-02-02-993, and the Ministry of Higher Education for grant FRGS Vot. 59001.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BI2219).

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 doi:10.1107/S1600536807038433

supplementary materials

Acta Cryst. (2007). E**63**, o3781 [**doi:10.1107/S1600536807038433**]

N-Butanoyl-*N*'-(4-nitrophenyl)thiourea

M. S. M. Yusof, Z. I. M. Saadum and B. M. Yamin

Comment

The title compound is similar to the previously reported compound *N*-(4-methylbenzoyl)-*N*'-(4-nitrophenyl)thiourea (Yusof *et al.*, 2006), except that the 4-methylbenzoyl group is replaced by a butanoyl group. The bond lengths and angles are comparable to those in that compound and to other thiourea derivatives (Yusof *et al.*, 2007*a*,*b*). The central thiourea fragment (S1/C5/N1/N2) and phenyl ring (C6–C7) are essentially planar with maximum deviation of 0.013 (1)Å for atom N1, and the dihedral angle between them is 5.27 (8)°. An intramolecular hydrogen bond, N2—H2A···O1, forms a *pseudo*-six-membered ring (O1···H2A/N2/C5/N1/C4), and a relatively short intramolecular C—H···S contact is also observed (H11A···S1 = 2.56 Å, C11···S1 = 3.222 (2) Å, C11—H11A···S1 = 128 °). In the crystal structure, the molecules are linked into two-dimensional networks parallel to the *bc* planes *via* intermolecular N—H···S and C—H···O interactions (Fig. 2 & Table 1).

Experimental

To a stirring acetone solution (75 ml) of butyrylchloride (2.0 g, 19 mmol) and ammoniumthiocyanate (1.43 g, 19 mmol), 4-nitroaniline (2.59 g, 19 mmol) in 45 ml of acetone was added dropwise. The solution 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 then dried under vacuum. Good quality crystals were obtained by recrystallization from DMSO. Yield 74% (2.03 g).

Refinement

All H atoms were visible in difference Fourier maps, but were placed geometrically at ideal positions then allowed to ride on their parent C or N atoms with C—H = 0.93–0.97 Å and N—H = 0.86 Å, and with U_{iso}(H)= 1.2(C/N) or 1.5U_{eq}(methyl C).

Figures

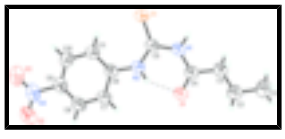

Fig. 1. The molecular structure of the title compound showing displacement ellipsoids at the 50% probability level for non-H atoms. The dashed line indicates an N—H···O hydrogen bond.

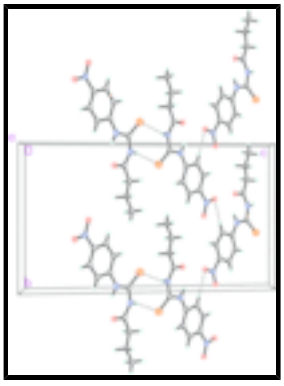

Fig. 2. Partial packing diagram, viewed down the *a*-axis. The dashed lines denote N—H···S and C—H···O interactions.

_N_-Butanoyl-_N_'-(4-nitrophenyl)thiourea

Crystal data

$C_{11}H_{13}N_3O_3S$	F_{000} = 560
M_r = 267.30	D_x = 1.367 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 894 reflections
a = 4.9002 (12) Å	θ = 1.9–26º
b = 12.385 (3) Å	μ = 0.25 mm^{-1}
c = 21.774 (5) Å	T = 293 (2) K
β = 100.727 (6)º	Block, light yellow
V = 1298.3 (5) Å^3	0.51 × 0.50 × 0.46 mm
Z = 4	

Data collection

Bruker SMART APEX CCD diffractometer	2540 independent reflections
Radiation source: fine-focus sealed tube	1933 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.018
T = 293(2) K	θ_{max} = 26.0º
ω scans	θ_{min} = 1.9º
Absorption correction: multi-scan (SADABS; Bruker, 2000)	h = −6→3
T_{min} = 0.882, T_{max} = 0.892	k = −15→14
7048 measured reflections	l = −26→26

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.043	H-atom parameters constrained
$wR(F^2)$ = 0.128	$w = 1/[\sigma^2(F_o^2) + (0.0696P)^2 + 0.2521P]$

	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} < 0.001$
2540 reflections	$\Delta\rho_{max} = 0.23$ e Å^{-3}
164 parameters	$\Delta\rho_{min} = -0.16$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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}
S1	0.82531 (12)	−0.85936 (4)	−0.46315 (2)	0.0606 (2)
O1	0.5256 (3)	−1.15131 (11)	−0.36784 (8)	0.0678 (4)
O2	−0.0653 (4)	−0.61661 (16)	−0.22970 (9)	0.0912 (6)
O3	0.1567 (5)	−0.50150 (15)	−0.27592 (10)	0.1057 (7)
N1	0.7852 (3)	−1.06019 (12)	−0.42783 (7)	0.0538 (4)
H1A	0.9045	−1.0698	−0.4517	0.065*
N2	0.5553 (3)	−0.93999 (12)	−0.37607 (7)	0.0535 (4)
H2A	0.5099	−0.9994	−0.3602	0.064*
N3	0.0939 (4)	−0.59385 (17)	−0.26469 (9)	0.0716 (5)
C1	0.8042 (7)	−1.4546 (2)	−0.43218 (16)	0.1014 (10)
H1B	0.6911	−1.5166	−0.4282	0.152*
H1C	0.9840	−1.4639	−0.4063	0.152*
H1D	0.8242	−1.4469	−0.4750	0.152*
C2	0.6687 (6)	−1.35530 (18)	−0.41193 (14)	0.0844 (8)
H2B	0.4791	−1.3512	−0.4347	0.101*
H2C	0.6622	−1.3613	−0.3678	0.101*
C3	0.8166 (5)	−1.25460 (17)	−0.42254 (12)	0.0687 (6)
H3A	0.8229	−1.2495	−0.4667	0.082*
H3B	1.0066	−1.2599	−0.4001	0.082*
C4	0.6927 (4)	−1.15281 (16)	−0.40304 (10)	0.0557 (5)
C5	0.7122 (4)	−0.95391 (15)	−0.41966 (8)	0.0478 (4)
C6	0.4504 (4)	−0.84770 (14)	−0.35126 (9)	0.0482 (4)
C7	0.2850 (4)	−0.86884 (16)	−0.30707 (9)	0.0558 (5)
H7A	0.2536	−0.9400	−0.2966	0.067*
C8	0.1682 (4)	−0.78694 (17)	−0.27893 (9)	0.0598 (5)
H8A	0.0564	−0.8015	−0.2498	0.072*

C9	0.2196 (4)	−0.68234 (17)	−0.29470 (9)	0.0567 (5)
C10	0.3827 (5)	−0.65870 (16)	−0.33746 (11)	0.0635 (6)
H10A	0.4146	−0.5873	−0.3472	0.076*
C11	0.4999 (4)	−0.74187 (16)	−0.36612 (10)	0.0611 (5)
H11A	0.6114	−0.7267	−0.3952	0.073*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0715 (4)	0.0573 (3)	0.0613 (4)	0.0000 (2)	0.0338 (3)	0.0062 (2)
O1	0.0797 (11)	0.0556 (8)	0.0808 (10)	−0.0014 (7)	0.0480 (9)	0.0016 (7)
O2	0.0923 (13)	0.0943 (13)	0.0985 (13)	0.0146 (10)	0.0478 (11)	−0.0184 (10)
O3	0.1409 (18)	0.0634 (12)	0.1246 (16)	0.0070 (11)	0.0552 (14)	−0.0201 (10)
N1	0.0552 (10)	0.0537 (9)	0.0599 (10)	−0.0003 (7)	0.0298 (8)	−0.0014 (7)
N2	0.0584 (10)	0.0490 (9)	0.0599 (9)	−0.0002 (7)	0.0285 (8)	0.0028 (7)
N3	0.0749 (13)	0.0690 (13)	0.0720 (12)	0.0105 (10)	0.0166 (10)	−0.0164 (10)
C1	0.127 (3)	0.0594 (15)	0.131 (2)	0.0039 (15)	0.058 (2)	−0.0052 (15)
C2	0.105 (2)	0.0579 (14)	0.105 (2)	−0.0031 (12)	0.0562 (17)	−0.0037 (12)
C3	0.0747 (15)	0.0552 (12)	0.0860 (16)	0.0019 (10)	0.0399 (12)	0.0003 (10)
C4	0.0574 (12)	0.0538 (11)	0.0607 (12)	−0.0012 (9)	0.0236 (10)	−0.0002 (8)
C5	0.0432 (10)	0.0551 (11)	0.0473 (10)	−0.0004 (8)	0.0143 (8)	0.0000 (8)
C6	0.0458 (10)	0.0505 (10)	0.0510 (10)	0.0013 (8)	0.0161 (8)	−0.0015 (8)
C7	0.0615 (13)	0.0548 (11)	0.0571 (11)	0.0016 (9)	0.0267 (10)	0.0043 (9)
C8	0.0606 (12)	0.0674 (13)	0.0564 (11)	0.0036 (10)	0.0241 (10)	−0.0014 (9)
C9	0.0532 (12)	0.0616 (12)	0.0567 (11)	0.0073 (9)	0.0140 (9)	−0.0097 (9)
C10	0.0697 (14)	0.0464 (11)	0.0781 (15)	−0.0015 (9)	0.0231 (12)	−0.0017 (9)
C11	0.0659 (13)	0.0543 (11)	0.0716 (13)	−0.0034 (9)	0.0346 (11)	−0.0003 (10)

Geometric parameters (Å, °)

S1—C5	1.6646 (19)	C2—H2B	0.970
O1—C4	1.221 (2)	C2—H2C	0.970
O2—N3	1.221 (3)	C3—C4	1.495 (3)
O3—N3	1.221 (3)	C3—H3A	0.970
N1—C4	1.380 (2)	C3—H3B	0.970
N1—C5	1.384 (2)	C6—C11	1.382 (3)
N1—H1A	0.860	C6—C7	1.393 (3)
N2—C5	1.339 (2)	C7—C8	1.365 (3)
N2—C6	1.402 (2)	C7—H7A	0.930
N2—H2A	0.860	C8—C9	1.375 (3)
N3—C9	1.469 (3)	C8—H8A	0.930
C1—C2	1.503 (3)	C9—C10	1.367 (3)
C1—H1B	0.960	C10—C11	1.383 (3)
C1—H1C	0.960	C10—H10A	0.930
C1—H1D	0.960	C11—H11A	0.930
C2—C3	1.482 (3)		
C4—N1—C5	128.95 (16)	H3A—C3—H3B	107.5
C4—N1—H1A	115.6	O1—C4—N1	122.71 (17)

C5—N1—H1A	115.5	O1—C4—C3	123.12 (17)
C5—N2—C6	132.70 (16)	N1—C4—C3	114.17 (17)
C5—N2—H2A	113.6	N2—C5—N1	114.19 (16)
C6—N2—H2A	113.7	N2—C5—S1	127.48 (14)
O2—N3—O3	123.7 (2)	N1—C5—S1	118.32 (13)
O2—N3—C9	118.4 (2)	C11—C6—C7	119.32 (17)
O3—N3—C9	117.9 (2)	C11—C6—N2	126.15 (17)
C2—C1—H1B	109.5	C7—C6—N2	114.52 (16)
C2—C1—H1C	109.5	C8—C7—C6	121.12 (18)
H1B—C1—H1C	109.5	C8—C7—H7A	119.4
C2—C1—H1D	109.5	C6—C7—H7A	119.4
H1B—C1—H1D	109.5	C7—C8—C9	118.45 (19)
H1C—C1—H1D	109.5	C7—C8—H8A	120.8
C3—C2—C1	112.8 (2)	C9—C8—H8A	120.8
C3—C2—H2B	109.0	C10—C9—C8	121.95 (18)
C1—C2—H2B	109.0	C10—C9—N3	119.3 (2)
C3—C2—H2C	109.0	C8—C9—N3	118.7 (2)
C1—C2—H2C	109.0	C9—C10—C11	119.49 (19)
H2B—C2—H2C	107.8	C9—C10—H10A	120.3
C2—C3—C4	115.40 (18)	C11—C10—H10A	120.3
C2—C3—H3A	108.4	C6—C11—C10	119.66 (18)
C4—C3—H3A	108.4	C6—C11—H11A	120.2
C2—C3—H3B	108.4	C10—C11—H11A	120.2
C4—C3—H3B	108.4		
C1—C2—C3—C4	−179.7 (2)	C6—C7—C8—C9	−0.6 (3)
C5—N1—C4—O1	−0.9 (4)	C7—C8—C9—C10	0.1 (3)
C5—N1—C4—C3	179.61 (19)	C7—C8—C9—N3	179.85 (18)
C2—C3—C4—O1	16.9 (4)	O2—N3—C9—C10	175.4 (2)
C2—C3—C4—N1	−163.6 (2)	O3—N3—C9—C10	−5.2 (3)
C6—N2—C5—N1	176.16 (18)	O2—N3—C9—C8	−4.4 (3)
C6—N2—C5—S1	−3.5 (3)	O3—N3—C9—C8	175.1 (2)
C4—N1—C5—N2	8.5 (3)	C8—C9—C10—C11	0.2 (3)
C4—N1—C5—S1	−171.81 (16)	N3—C9—C10—C11	−179.57 (19)
C5—N2—C6—C11	−2.9 (3)	C7—C6—C11—C10	−0.6 (3)
C5—N2—C6—C7	178.0 (2)	N2—C6—C11—C10	−179.7 (2)
C11—C6—C7—C8	0.9 (3)	C9—C10—C11—C6	0.1 (3)
N2—C6—C7—C8	−179.92 (19)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2A···O1	0.86	1.89	2.629 (2)	143
N1—H1A···S1^{i}	0.86	2.62	3.4579 (18)	164
C7—H7A···O2^{ii}	0.93	2.48	3.397 (3)	168

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

Fig. 1

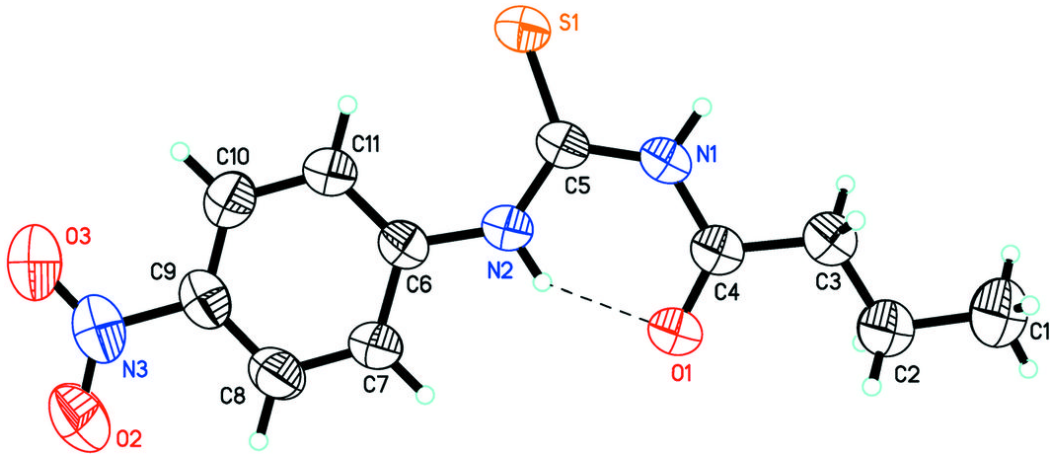

Fig. 2