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1-Ethyl-1-methyl-3-(2-nitrobenzoyl)thiourea

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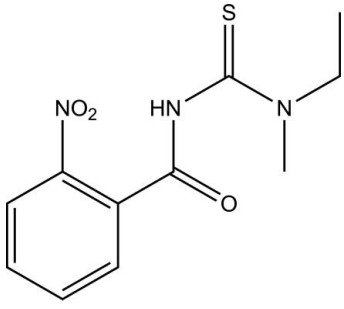
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.043; wR factor = 0.124; data-to-parameter ratio = 13.6.

In the title compound, $C_{11}H_{13}N_3O_3S$, the benzene ring is twisted relative to the amidic fragment, forming a dihedral angle of 27.26 (9)°. The thiono and carbonyl groups are *trans* with respect to the C—N bond. Intermolecular N—H···S and C—H···O hydrogen bonds link the molecules in the crystal structure.

Related literature

For the synthesis, see: Al-abbasi *et al.* (2010). For related structures and background references, see: Shanmuga Sundara Raj *et al.* (1999); Arslan *et al.* (2003); Al-abbasi & Kassim (2011). For standard bond lengths, see: Allen *et al.* (1987) and for bond lengths in other substituted thioureas, see: Nasir *et al.* (2011); Pérez *et al.* (2011).



Experimental

Crystal data

$C_{11}H_{13}N_3O_3S$
M_r = 267.30
Monoclinic, $P2_1/n$
a = 11.447 (2) Å
b = 7.8664 (15) Å
c = 15.159 (3) Å
β = 107.128 (4)°
V = 1304.5 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 298 K
0.55 × 0.38 × 0.21 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.874, T_{max} = 0.949
7105 measured reflections
2294 independent reflections
1971 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.124
S = 1.06
2294 reflections
169 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···S1^{i}	0.85 (2)	2.55 (2)	3.3828 (18)	167 (2)
C6—H6···O3^{ii}	0.93	2.41	3.317 (3)	164

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; 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: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JH2299).

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supporting information

Acta Cryst. (2011). E**67**, o1840 [doi:10.1107/S1600536811024652]

1-Ethyl-1-methyl-3-(2-nitrobenzoyl)thiourea

Aisha A. Al-abbasi and Mohammad B. Kassim

S1. Comment

The title compound, I, is a thiourea derivative analogous to our previously reported compounds (Al-abbasi & Kassim, 2011). Bond distances are similar to those usually found in other substituted thioureas [Nasir *et al.* (2011) & Pérez *et al.* (2011)]. The C–S and C–O exhibited the expected double-bond character. However, the C–N bond lengths are intermediate between a single and double, indicating a partial electron delocalization in the O1/C7/N1/C8/S1 fragment.

The phenyl ring is twisted due to the presence of the nitro group (O2O3N3) in *ortho* position. A rotation around C1—C7 bond makes the oxygen atom (O1) perpendicular to the phenyl ring mean planes and the torsion angles of C2C1C701 and C6C1C701 are -95.5 (2) and 86.5 (2)°, respectively. The dihedral angle between the mean planes of the thiourea (S1/N1/N2/C8/C9) and the phenyl ring (C1/C2/C3/C4/C5/C6/) plane is 27.56 (10)°. Other bond lengths and angles are in normal ranges (Allen *et al.* 1987).

The crystal structure is stabilized by the intermolecular N1—H1A···S1 and C5—H5A···O3 hydrogen bonds linking the molecules into a dimer resulting in a channel along [101] (Fig. 2).

S2. Experimental

The title compound was prepared according to a previously reported procedure (Al-abbasi *et al.*, 2010). A very pale browon colour crystal, suitable for X-ray crystallography, was obtained by a slow evaporation from ethanol solution at room temperature (yield 78%).

S3. Refinement

Hydrogen atom of the amide group was determined from the diffrence Fourrier map and N—H was initially fixed at 0.86(0.01) Å and allowed to be refined on the parent N atom with U_{iso}(H) = 1.2U_{eq}(N). All other H atoms were postioned geometrically with C—H bond lengths in the range 0.93 - 0.97 Å and refined in the riding model approximation with U_{iso}(H)=1.2U_{eq}(C,*N*), except for methyl group where U_{iso}(H)= 1.5U_{eq}(C).

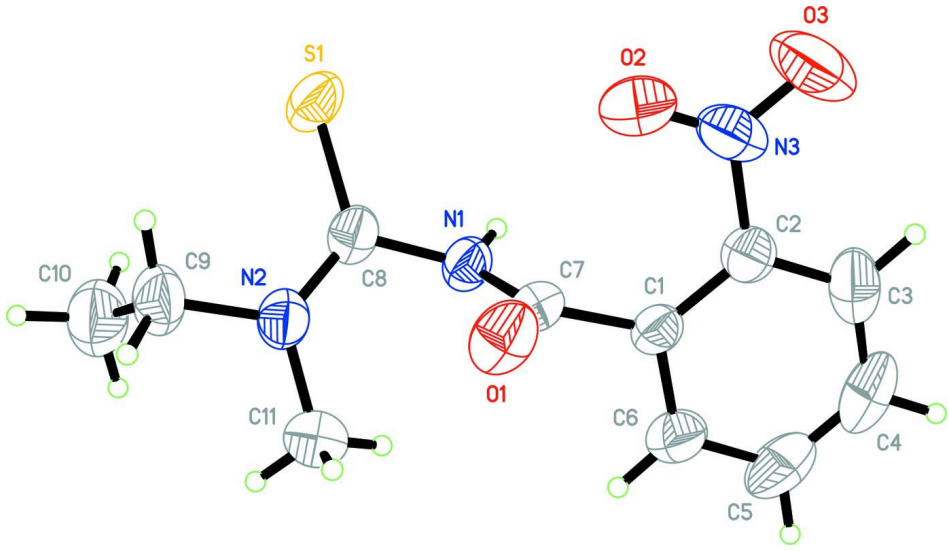

Figure 1

The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level.

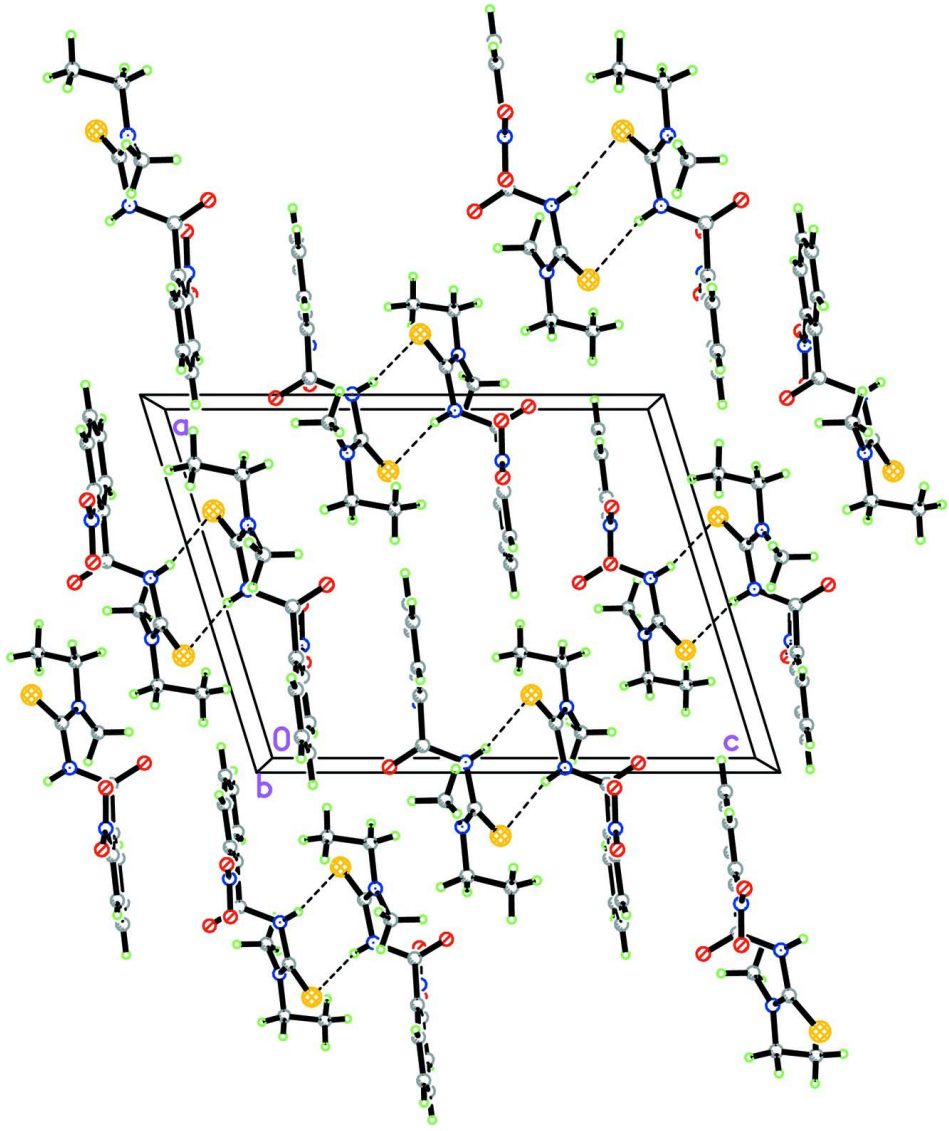

Figure 2

A packing diagram of the title compound viewed down the *a*-axis showing the intermolecular hydrogen bonds N1—H1A···S1 (*-x + 1, -y, -z*) and C6—H6···O3 (*x, y + 1, z*).

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Crystal data

$C_{11}H_{13}N_3O_3S$
$M_r = 267.30$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 11.447\ (2)$ Å
$b = 7.8664\ (15)$ Å
$c = 15.159\ (3)$ Å
$\beta = 107.128\ (4)°$
$V = 1304.5\ (4)$ Å^3
$Z = 4$
$F(000) = 560$
$D_x = 1.361$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4015 reflections
$\theta = 2.0–25.0°$
$\mu = 0.25$ mm^{-1}
$T = 298$ K
Block, brown
0.55 × 0.38 × 0.21 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.874, T_{max} = 0.949
7105 measured reflections
2294 independent reflections
1971 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 25.0°, θ_{min} = 2.0°
h = −13→13
k = −7→9
l = −15→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.124
S = 1.06
2294 reflections
169 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.065P)^2 + 0.531P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
S1	0.69163 (5)	0.06431 (8)	0.07382 (4)	0.0575 (2)
O1	0.49702 (13)	0.2128 (2)	0.24893 (10)	0.0608 (4)
O2	0.44396 (18)	−0.1541 (2)	0.17973 (16)	0.0811 (6)
O3	0.2858 (2)	−0.3156 (2)	0.14610 (17)	0.0984 (7)
N1	0.48480 (14)	0.1814 (2)	0.09706 (11)	0.0435 (4)
N2	0.65025 (15)	0.3643 (2)	0.14053 (13)	0.0528 (5)
N3	0.3344 (2)	−0.1771 (2)	0.15678 (14)	0.0612 (5)
C1	0.30487 (17)	0.1334 (2)	0.14682 (13)	0.0401 (4)
C2	0.25371 (19)	−0.0265 (3)	0.14295 (14)	0.0459 (5)
C3	0.1301 (2)	−0.0513 (4)	0.12710 (16)	0.0636 (7)
H3	0.0984	−0.1604	0.1258	0.076*
C4	0.0545 (2)	0.0879 (4)	0.11328 (18)	0.0731 (8)
H4	−0.0291	0.0735	0.1026	0.088*
C5	0.1024 (2)	0.2479 (4)	0.11526 (19)	0.0728 (8)
H5	0.0507	0.3418	0.1050	0.087*

C6	0.2264 (2)	0.2709 (3)	0.13230 (16)	0.0558 (6)
H6	0.2577	0.3803	0.1341	0.067*
C7	0.43942 (17)	0.1743 (2)	0.17127 (14)	0.0433 (5)
C8	0.60936 (17)	0.2138 (3)	0.10694 (13)	0.0441 (5)
C9	0.7795 (2)	0.4114 (3)	0.16004 (17)	0.0607 (6)
H9A	0.8292	0.3093	0.1710	0.073*
H9B	0.8039	0.4801	0.2156	0.073*
C10	0.8020 (3)	0.5099 (4)	0.0808 (2)	0.0793 (8)
H10A	0.7897	0.4366	0.0282	0.119*
H10B	0.8844	0.5518	0.0987	0.119*
H10C	0.7461	0.6038	0.0651	0.119*
C11	0.5729 (2)	0.5026 (3)	0.1556 (2)	0.0737 (8)
H11A	0.4905	0.4853	0.1175	0.111*
H11B	0.6028	0.6090	0.1400	0.111*
H11C	0.5747	0.5042	0.2193	0.111*
H1A	0.4498 (18)	0.123 (3)	0.0494 (11)	0.051 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0395 (3)	0.0708 (4)	0.0647 (4)	0.0004 (2)	0.0194 (3)	−0.0241 (3)
O1	0.0506 (9)	0.0857 (12)	0.0461 (9)	−0.0004 (8)	0.0142 (7)	−0.0123 (8)
O2	0.0695 (12)	0.0584 (11)	0.1181 (16)	0.0213 (9)	0.0318 (11)	0.0114 (10)
O3	0.134 (2)	0.0423 (10)	0.1218 (18)	−0.0117 (11)	0.0429 (15)	−0.0064 (10)
N1	0.0362 (8)	0.0513 (10)	0.0453 (9)	−0.0046 (7)	0.0156 (7)	−0.0126 (8)
N2	0.0415 (9)	0.0572 (11)	0.0618 (11)	−0.0084 (8)	0.0185 (8)	−0.0138 (9)
N3	0.0866 (15)	0.0377 (10)	0.0639 (12)	0.0017 (10)	0.0295 (11)	0.0017 (8)
C1	0.0402 (10)	0.0417 (10)	0.0428 (10)	0.0035 (8)	0.0190 (8)	−0.0007 (8)
C2	0.0504 (11)	0.0470 (11)	0.0449 (11)	0.0001 (9)	0.0209 (9)	0.0002 (8)
C3	0.0608 (14)	0.0764 (17)	0.0602 (14)	−0.0244 (13)	0.0279 (12)	−0.0070 (12)
C4	0.0389 (12)	0.118 (2)	0.0658 (16)	−0.0025 (14)	0.0213 (11)	−0.0029 (15)
C5	0.0510 (14)	0.090 (2)	0.0805 (18)	0.0280 (14)	0.0242 (12)	0.0086 (14)
C6	0.0536 (12)	0.0485 (12)	0.0698 (14)	0.0113 (10)	0.0252 (11)	0.0041 (10)
C7	0.0405 (10)	0.0429 (11)	0.0497 (11)	0.0049 (8)	0.0181 (9)	−0.0035 (8)
C8	0.0367 (10)	0.0560 (12)	0.0400 (10)	−0.0039 (9)	0.0122 (8)	−0.0071 (8)
C9	0.0448 (12)	0.0725 (15)	0.0638 (14)	−0.0165 (11)	0.0145 (10)	−0.0187 (12)
C10	0.0656 (16)	0.0856 (19)	0.090 (2)	−0.0134 (14)	0.0287 (14)	−0.0004 (16)
C11	0.0675 (16)	0.0533 (14)	0.107 (2)	−0.0033 (12)	0.0356 (15)	−0.0200 (14)

Geometric parameters (Å, º)

S1—C8	1.674 (2)	C3—H3	0.9300
O1—C7	1.206 (2)	C4—C5	1.370 (4)
O2—N3	1.212 (3)	C4—H4	0.9300
O3—N3	1.212 (3)	C5—C6	1.378 (3)
N1—C7	1.372 (2)	C5—H5	0.9300
N1—C8	1.412 (2)	C6—H6	0.9300
N1—H1A	0.849 (10)	C9—C10	1.514 (4)

N2—C8	1.319 (3)	C9—H9A	0.9700
N2—C11	1.462 (3)	C9—H9B	0.9700
N2—C9	1.469 (3)	C10—H10A	0.9600
N3—C2	1.479 (3)	C10—H10B	0.9600
C1—C2	1.381 (3)	C10—H10C	0.9600
C1—C6	1.382 (3)	C11—H11A	0.9600
C1—C7	1.509 (3)	C11—H11B	0.9600
C2—C3	1.378 (3)	C11—H11C	0.9600
C3—C4	1.372 (4)		

C7—N1—C8	122.31 (16)	C1—C6—H6	119.6
C7—N1—H1A	118.6 (15)	O1—C7—N1	124.04 (18)
C8—N1—H1A	113.3 (15)	O1—C7—C1	121.22 (17)
C8—N2—C11	124.45 (18)	N1—C7—C1	114.38 (17)
C8—N2—C9	121.75 (18)	N2—C8—N1	115.81 (17)
C11—N2—C9	113.66 (19)	N2—C8—S1	125.42 (15)
O3—N3—O2	124.6 (2)	N1—C8—S1	118.75 (15)
O3—N3—C2	117.3 (2)	N2—C9—C10	111.5 (2)
O2—N3—C2	118.12 (18)	N2—C9—H9A	109.3
C2—C1—C6	117.29 (18)	C10—C9—H9A	109.3
C2—C1—C7	126.44 (17)	N2—C9—H9B	109.3
C6—C1—C7	116.16 (18)	C10—C9—H9B	109.3
C3—C2—C1	122.5 (2)	H9A—C9—H9B	108.0
C3—C2—N3	118.5 (2)	C9—C10—H10A	109.5
C1—C2—N3	118.97 (18)	C9—C10—H10B	109.5
C4—C3—C2	118.9 (2)	H10A—C10—H10B	109.5
C4—C3—H3	120.6	C9—C10—H10C	109.5
C2—C3—H3	120.6	H10A—C10—H10C	109.5
C5—C4—C3	120.0 (2)	H10B—C10—H10C	109.5
C5—C4—H4	120.0	N2—C11—H11A	109.5
C3—C4—H4	120.0	N2—C11—H11B	109.5
C4—C5—C6	120.5 (2)	H11A—C11—H11B	109.5
C4—C5—H5	119.8	N2—C11—H11C	109.5
C6—C5—H5	119.8	H11A—C11—H11C	109.5
C5—C6—C1	120.9 (2)	H11B—C11—H11C	109.5
C5—C6—H6	119.6		

C6—C1—C2—C3	−1.2 (3)	C8—N1—C7—O1	8.5 (3)
C7—C1—C2—C3	174.71 (19)	C8—N1—C7—C1	−178.37 (17)
C6—C1—C2—N3	179.13 (19)	C2—C1—C7—O1	−95.5 (3)
C7—C1—C2—N3	−5.0 (3)	C6—C1—C7—O1	80.5 (3)
O3—N3—C2—C3	5.7 (3)	C2—C1—C7—N1	91.2 (2)
O2—N3—C2—C3	−172.8 (2)	C6—C1—C7—N1	−92.9 (2)
O3—N3—C2—C1	−174.6 (2)	C11—N2—C8—N1	−8.5 (3)
O2—N3—C2—C1	6.9 (3)	C9—N2—C8—N1	176.06 (19)
C1—C2—C3—C4	1.0 (3)	C11—N2—C8—S1	170.1 (2)
N3—C2—C3—C4	−179.3 (2)	C9—N2—C8—S1	−5.4 (3)
C2—C3—C4—C5	0.1 (4)	C7—N1—C8—N2	−63.8 (3)

C3—C4—C5—C6	−0.9 (4)	C7—N1—C8—S1	117.53 (18)
C4—C5—C6—C1	0.6 (4)	C8—N2—C9—C10	96.1 (3)
C2—C1—C6—C5	0.4 (3)	C11—N2—C9—C10	−79.8 (3)
C7—C1—C6—C5	−175.9 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···S1^{i}	0.85 (2)	2.55 (2)	3.3828 (18)	167 (2)
C6—H6···O3^{ii}	0.93	2.41	3.317 (3)	164

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