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N-[*N*-(4-Methylbenzoyl)carbamothioyl]-glycine

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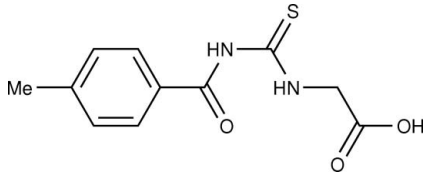
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.038; wR factor = 0.107; data-to-parameter ratio = 14.9.

In the title molecule, $C_{11}H_{12}N_2O_3S$, all bond lengths and angles are normal. Intramolecular N—H···O and C—H···S hydrogen bonds influence the molecular conformation. The crystal packing exhibits an extensive network formed by intermolecular O—H···S, N—H···O and C—H···O hydrogen bonds.

Related literature

For a related crystal structure, see: Yusof *et al.* (2006). For normal ranges of molecular bond lengths, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{11}H_{12}N_2O_3S$
M_r = 252.29
Monoclinic, $P2_1/n$
a = 11.664 (3) Å
b = 8.590 (2) Å
c = 12.962 (3) Å
β = 113.917 (4)°
V = 1187.2 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.27 mm^{-1}
T = 298 (2) K
0.50 × 0.48 × 0.21 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.877, T_{max} = 0.945
6388 measured reflections
2322 independent reflections
2018 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.107
S = 1.06
2322 reflections
156 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2B···O1	0.86	1.97	2.639 (2)	133
C10—H10B···S1	0.97	2.70	3.0413 (19)	101
O2—H2A···S1^{i}	0.82	2.33	3.1458 (16)	172
N1—H1A···O3^{ii}	0.86	2.17	2.964 (2)	153
N2—H2B···O3^{iii}	0.86	2.54	3.0481 (19)	119
C7—H7A···O1^{iv}	0.96	2.50	3.455 (4)	173

Symmetry codes: (i) $x+\frac{1}{2}, -y+\frac{3}{2}, z+\frac{1}{2}$; (ii) $x-\frac{1}{2}, -y+\frac{3}{2}, z-\frac{1}{2}$; (iii) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{5}{2}$; (iv) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$.

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: CV2280).

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supplementary materials

Acta Cryst. (2007). E**63**, o3591 [**doi:10.1107/S1600536807035088**]

N-[*N*-(4-Methylbenzoyl)carbamothioyl]glycine

M. S. M. Yusof, R. Roslan, M. A. Kadir and B. M. Yamin

Comment

The title compound, (I), is analogous to *N*-(4-Methylbenzoyl)-*N*'-(4-nitrophenyl)thiourea, (II) (Yusof *et al.*, 2006), except that the 4-nitrophenyl group is replaced by amino-acetic acid group (Fig. 1). The molecule of (I) maintains its *trans-cis* configuration with respect to the position of the 4-methylbenzoyl and acetic acid groups relative to the thiono S1 atom across the C8—N1 and C9—N2 bonds, respectively. The bond lengths and angles are in normal ranges (Allen *et al.*, 1987) and are comparable to those in (II). The central thiourea, (S1/C9/N1/N2), phenyl ring (C1—C6) and amino-acetic acid (C10/C11/O2/O3/N2) fragments are essentially planar each with maximum deviation of 0.031 (2)° for atom C10 from the least square planes. The dihedral angles between the central thiourea and phenyl ring fragments is 8.23 (8)°. There are two intramolecular hydrogen bonds, N2—H2···O1 and C10—H10···S1 (Table 1), and as a result, a pseudo-six- and five-membered rings (O1···H2—N2—C9—N1—C8—O1 and H10B···S1—C9—N2—C10—H10B) are formed. The crystal packing exhibits extensive network formed by intermolecular O—H···S, N—H···O and C—H···O hydrogen bonds (Table 1).

Experimental

An equimolar amount of glycine (1.0 g, 0.01 mol) in 20 ml acetone was added dropwise into a stirring acetone solution (75 ml) containing 4-methylbenzoyl chloride (2.36 g, 0.01 mol) and ammonium thiocyanate (0.98 g, 0.01 mol). The solution mixture was refluxed for 6 h. The resulting solution was poured into a beaker containing some ice blocks. The white precipitate was filtered and washed with distilled water and cold ethanol before dried under vacuum. Good quality crystals were obtained by recrystallization from methanol. Yield 77% (2.05 g). MP 375.2–376.4 K.

Refinement

After their location in the difference map, all H-atoms were fixed geometrically at ideal positions (O—H, N—H and C—H of 0.82, 0.86 and 0.93–0.96 Å, respectively) and allowed to ride on the parent atoms with U_{iso}(H)= 1.2–1.5U_{eq} of the parent atom.

Figures

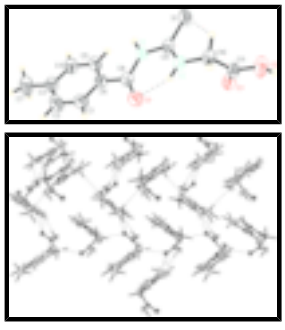

Fig. 1. Molecular structure of the title compound, (I), with the 50% probability displacement ellipsoids. The dashed line indicates intramolecular N—H···O and C—H···S hydrogen bonds.

N-[*N*-(4-Methylbenzoyl)carbamothioyl]glycine

Crystal data

$C_{11}H_{12}N_2O_3S$	F_{000} = 528
M_r = 252.29	D_x = 1.412 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 11.664 (3) Å	Cell parameters from 3055 reflections
b = 8.590 (2) Å	θ = 1.9–26.0º
c = 12.962 (3) Å	μ = 0.27 mm^{-1}
β = 113.917 (4)º	T = 298 (2) K
V = 1187.2 (5) Å^3	Block, colourless
Z = 4	0.50 × 0.48 × 0.21 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer	2322 independent reflections
Radiation source: fine-focus sealed tube	2018 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.017
Detector resolution: 83.66 pixels mm^{-1}	θ_{max} = 26.0º
T = 298(2) K	θ_{min} = 2.0º
ω scans	h = −14→8
Absorption correction: multi-scan (SADABS; Bruker, 2000)	k = −10→10
T_{min} = 0.877, T_{max} = 0.945	l = −15→15
6388 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.038	H-atom parameters constrained
$wR(F^2)$ = 0.107	$w = 1/[\sigma^2(F_o^2) + (0.0613P)^2 + 0.2581P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.06	$(\Delta/\sigma)_{max} < 0.001$
2322 reflections	$\Delta\rho_{max}$ = 0.20 e Å^{-3}
156 parameters	$\Delta\rho_{min}$ = −0.22 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	0.01912 (5)	0.77866 (6)	1.17790 (4)	0.05108 (18)
O1	0.23600 (14)	0.50912 (19)	1.01638 (11)	0.0661 (4)
O2	0.29873 (13)	0.54526 (17)	1.48607 (9)	0.0601 (4)
H2A	0.3527	0.5997	1.5328	0.090*
O3	0.34152 (12)	0.69059 (14)	1.36404 (10)	0.0477 (3)
N1	0.08679 (13)	0.67815 (16)	1.01690 (11)	0.0402 (3)
H1A	0.0310	0.7432	0.9756	0.048*
N2	0.16263 (13)	0.53866 (15)	1.18306 (10)	0.0399 (3)
H2B	0.2001	0.4820	1.1515	0.048*
C1	0.17221 (17)	0.5590 (2)	0.78550 (15)	0.0505 (4)
H1	0.2181	0.4699	0.8178	0.061*
C2	0.14413 (19)	0.5940 (2)	0.67371 (16)	0.0564 (5)
H2	0.1723	0.5281	0.6320	0.068*
C3	0.07530 (18)	0.7243 (2)	0.62259 (15)	0.0492 (4)
C4	0.03829 (19)	0.8234 (2)	0.68810 (15)	0.0501 (4)
H4	−0.0063	0.9134	0.6560	0.060*
C5	0.06646 (17)	0.79070 (19)	0.79981 (14)	0.0441 (4)
H5	0.0413	0.8591	0.8423	0.053*
C6	0.13205 (15)	0.6564 (2)	0.84968 (13)	0.0405 (4)
C7	0.0398 (3)	0.7588 (3)	0.49940 (18)	0.0707 (6)
H7A	0.1037	0.8216	0.4914	0.106*
H7B	−0.0386	0.8137	0.4694	0.106*
H7C	0.0316	0.6628	0.4590	0.106*
C8	0.15811 (15)	0.6075 (2)	0.96716 (14)	0.0426 (4)
C9	0.09453 (15)	0.65652 (18)	1.12535 (13)	0.0369 (4)
C10	0.17812 (16)	0.49900 (18)	1.29587 (13)	0.0399 (4)
H10A	0.1966	0.3888	1.3083	0.048*
H10B	0.1002	0.5187	1.3037	0.048*
C11	0.28158 (15)	0.59043 (18)	1.38379 (12)	0.0371 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0641 (3)	0.0556 (3)	0.0328 (2)	0.0194 (2)	0.0189 (2)	0.00378 (18)
O1	0.0655 (8)	0.0874 (10)	0.0461 (7)	0.0351 (8)	0.0232 (7)	0.0126 (7)
O2	0.0708 (9)	0.0704 (9)	0.0297 (6)	−0.0245 (7)	0.0105 (6)	0.0026 (6)
O3	0.0497 (7)	0.0497 (7)	0.0377 (6)	−0.0116 (6)	0.0117 (5)	0.0022 (5)
N1	0.0445 (8)	0.0444 (7)	0.0285 (6)	0.0070 (6)	0.0117 (6)	0.0026 (6)
N2	0.0455 (8)	0.0389 (7)	0.0300 (7)	0.0043 (6)	0.0098 (6)	−0.0009 (5)
C1	0.0518 (11)	0.0563 (10)	0.0470 (10)	0.0065 (8)	0.0238 (8)	−0.0004 (8)
C2	0.0664 (12)	0.0646 (12)	0.0499 (10)	−0.0028 (10)	0.0355 (10)	−0.0094 (9)
C3	0.0551 (11)	0.0573 (11)	0.0389 (9)	−0.0155 (9)	0.0227 (8)	−0.0042 (8)
C4	0.0576 (11)	0.0488 (10)	0.0458 (10)	−0.0041 (8)	0.0230 (9)	0.0053 (8)
C5	0.0515 (10)	0.0435 (9)	0.0411 (9)	−0.0051 (7)	0.0227 (8)	−0.0039 (7)
C6	0.0369 (8)	0.0485 (9)	0.0349 (8)	−0.0053 (7)	0.0132 (7)	−0.0030 (7)
C7	0.0918 (17)	0.0805 (15)	0.0445 (11)	−0.0150 (13)	0.0325 (12)	−0.0004 (10)
C8	0.0386 (9)	0.0497 (9)	0.0368 (9)	0.0018 (7)	0.0125 (7)	−0.0024 (7)
C9	0.0380 (8)	0.0381 (8)	0.0293 (7)	−0.0035 (7)	0.0081 (6)	−0.0025 (6)
C10	0.0447 (9)	0.0377 (8)	0.0318 (8)	−0.0024 (7)	0.0100 (7)	0.0030 (6)
C11	0.0396 (8)	0.0369 (8)	0.0314 (8)	0.0033 (7)	0.0109 (7)	0.0029 (6)

Geometric parameters (Å, °)

S1—C9	1.6806 (17)	C2—H2	0.9300
O1—C8	1.215 (2)	C3—C4	1.389 (3)
O2—C11	1.3164 (19)	C3—C7	1.507 (3)
O2—H2A	0.8200	C4—C5	1.378 (2)
O3—C11	1.200 (2)	C4—H4	0.9300
N1—C8	1.382 (2)	C5—C6	1.390 (2)
N1—C9	1.384 (2)	C5—H5	0.9300
N1—H1A	0.8600	C6—C8	1.488 (2)
N2—C9	1.317 (2)	C7—H7A	0.9600
N2—C10	1.440 (2)	C7—H7B	0.9600
N2—H2B	0.8600	C7—H7C	0.9600
C1—C2	1.383 (3)	C10—C11	1.502 (2)
C1—C6	1.389 (2)	C10—H10A	0.9700
C1—H1	0.9300	C10—H10B	0.9700
C2—C3	1.381 (3)		
C11—O2—H2A	109.5	C1—C6—C8	117.67 (16)
C8—N1—C9	127.66 (14)	C5—C6—C8	123.78 (15)
C8—N1—H1A	116.2	C3—C7—H7A	109.5
C9—N1—H1A	116.2	C3—C7—H7B	109.5
C9—N2—C10	123.87 (14)	H7A—C7—H7B	109.5
C9—N2—H2B	118.1	C3—C7—H7C	109.5
C10—N2—H2B	118.1	H7A—C7—H7C	109.5
C2—C1—C6	120.17 (18)	H7B—C7—H7C	109.5
C2—C1—H1	119.9	O1—C8—N1	121.58 (15)

C6—C1—H1	119.9	O1—C8—C6	122.21 (15)
C3—C2—C1	121.59 (17)	N1—C8—C6	116.20 (14)
C3—C2—H2	119.2	N2—C9—N1	117.14 (14)
C1—C2—H2	119.2	N2—C9—S1	122.96 (12)
C2—C3—C4	117.90 (17)	N1—C9—S1	119.90 (12)
C2—C3—C7	121.61 (18)	N2—C10—C11	112.22 (13)
C4—C3—C7	120.49 (19)	N2—C10—H10A	109.2
C5—C4—C3	121.12 (18)	C11—C10—H10A	109.2
C5—C4—H4	119.4	N2—C10—H10B	109.2
C3—C4—H4	119.4	C11—C10—H10B	109.2
C4—C5—C6	120.65 (16)	H10A—C10—H10B	107.9
C4—C5—H5	119.7	O3—C11—O2	124.22 (15)
C6—C5—H5	119.7	O3—C11—C10	124.84 (14)
C1—C6—C5	118.52 (16)	O2—C11—C10	110.93 (14)
C6—C1—C2—C3	−0.6 (3)	C1—C6—C8—O1	17.6 (3)
C1—C2—C3—C4	2.2 (3)	C5—C6—C8—O1	−164.65 (18)
C1—C2—C3—C7	−177.24 (19)	C1—C6—C8—N1	−161.14 (16)
C2—C3—C4—C5	−1.7 (3)	C5—C6—C8—N1	16.6 (2)
C7—C3—C4—C5	177.80 (18)	C10—N2—C9—N1	−178.83 (14)
C3—C4—C5—C6	−0.5 (3)	C10—N2—C9—S1	1.4 (2)
C2—C1—C6—C5	−1.7 (3)	C8—N1—C9—N2	−11.8 (2)
C2—C1—C6—C8	176.21 (16)	C8—N1—C9—S1	168.04 (14)
C4—C5—C6—C1	2.2 (3)	C9—N2—C10—C11	−84.95 (19)
C4—C5—C6—C8	−175.53 (16)	N2—C10—C11—O3	3.3 (2)
C9—N1—C8—O1	3.0 (3)	N2—C10—C11—O2	−176.19 (14)
C9—N1—C8—C6	−178.23 (15)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2B···O1	0.86	1.97	2.639 (2)	133
C10—H10B···S1	0.97	2.70	3.0413 (19)	101
O2—H2A···S1^{i}	0.82	2.33	3.1458 (16)	172
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C7—H7A···O1^{iv}	0.96	2.50	3.455 (4)	173

Symmetry codes: (i) *x*+1/2, −*y*+3/2, *z*+1/2; (ii) *x*−1/2, −*y*+3/2, *z*−1/2; (iii) −*x*+1/2, *y*−1/2, −*z*+5/2; (iv) −*x*+1/2, *y*+1/2, −*z*+3/2.

Fig. 1

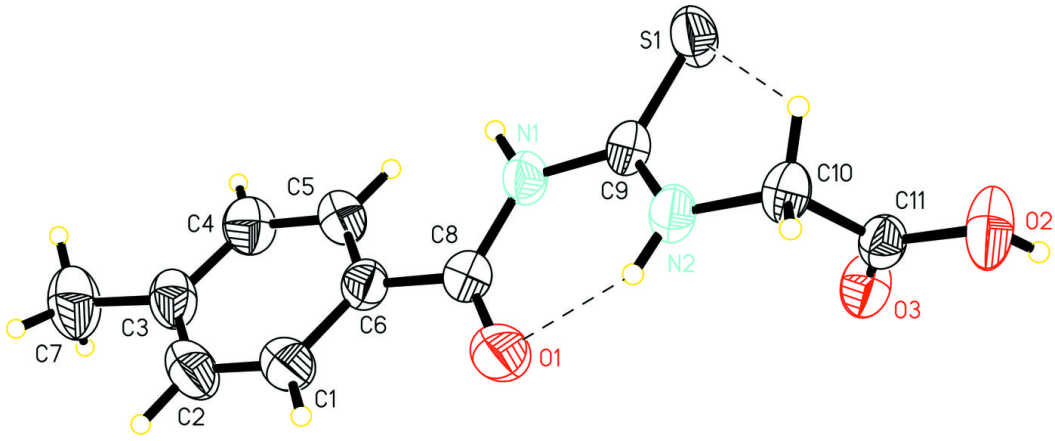

Fig. 2