Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-Acetyl-1-(2-methylphenyl)thiourea

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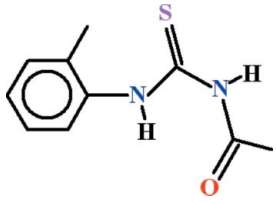
Received 17 March 2012; accepted 18 March 2012

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.037; wR factor = 0.088; data-to-parameter ratio = 14.0.

In the title compound, $C_{10}H_{12}N_2OS$, the toluene and the *N*-carbamothioylacetamide units are oriented at dihedral angle of 78.75 (5)°. An intramolecular N—H···O hydrogen bond generates an $S(6)$ ring. In the crystal, molecules are linked into [101] chains by pairs of N—H···S hydrogen bonds [which generate $R_2^2(8)$ loops] and pairs of O—H···O hydrogen bonds [which generate $R_2^2(4)$ loops]. The two motifs alternate in the chain.

Related literature

For related structures, see: Shahwar *et al.* (2012). For graph–set notation, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{10}H_{12}N_2OS$
M_r = 208.28
Monoclinic, $P2_1/c$
a = 5.0444 (2) Å
b = 20.7019 (9) Å
c = 9.9464 (4) Å
β = 95.116 (2)°
V = 1034.55 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.28 mm^{-1}
T = 296 K
0.35 × 0.15 × 0.13 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.915, T_{max} = 0.938
7696 measured reflections
1812 independent reflections
1512 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.088
S = 1.17
1812 reflections
129 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1	0.86	1.99	2.664 (2)	135
N1—H1···O1^{i}	0.86	2.50	3.172 (2)	135
N2—H2···S1^{ii}	0.86	2.52	3.3747 (17)	171

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

The authors acknowledge the provision of funds for the purchase of diffractometer and encouragement by Dr Muhammad Akram Chaudhary, Vice Chancellor, University of Sargodha, Pakistan. They also acknowledge the technical support provided by Syed Muhammad Hussain Rizvi of Bana International, Karachi, Pakistan.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6692).

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supplementary materials

Acta Cryst. (2012). E**68**, o1160 [doi:10.1107/S1600536812011658]

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Comment

The title compound **I** (Fig. 1) has been synthesized in continuation of our efforts to find new enzyme inhibitors.

The crystal structures of *N*-(phenylcarbamothioyl)acetamide (Shahwar *et al.*, 2012) has been published which is related to the title compound (I).

In (I), the toluene group A (C1–C7) and the *N*-carbamothioylacetamide moiety B (N1/C8/S1/N2/C9/O1/C10) are planar with r. m. s. deviation of 0.0058 Å and 0.0278 Å, respectively. The dihedral angle between A/B is 78.75 (5)°. There exist classical intramolecular H–bonding of N—H···O type (Table 1, Fig. 1) with *S*(6) ring motif (Bernstein *et al.*, 1995). The molecules are dimerized due to N—H···S type of hydrogen bonds with $R_2^2(8)$ ring motifs (Table 1, Fig. 2). The dimers are interlinked from S(6) ring motifs due to strong N—H···O H–bondings (Table 1, Fig. 2) with centrosymmetric four membered ring motif (—O···H···O···H···O—) (Table 1, Fig. 2). The polymeric chains extend along the base vector [101].

Experimental

The title compound (I) was synthesized by adding (0.1 mol, 7.13 ml) of acetylchloride dropwise to a stirred solution of KSCN (0.11 mol) in dry acetone (50 ml), followed by slow addition of toluidine (0.1 mol) in dry acetone (25 ml). The mixture was refluxed for 5–10 min, then poured on ice cooled water, which resulted in crude precipitate. Recrystallization of the precipitate in ethylacetate yielded colourless needles.

Refinement

The H-atoms were positioned geometrically (C—H = 0.93–0.96 Å, N—H = 0.86 Å) and refined as riding with U_{iso}(H) = xU_{eq}(C, N), where x = 1.5 for methyl groups and x = 1.2 for other H atoms.

Computing details

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT* (Bruker, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009).

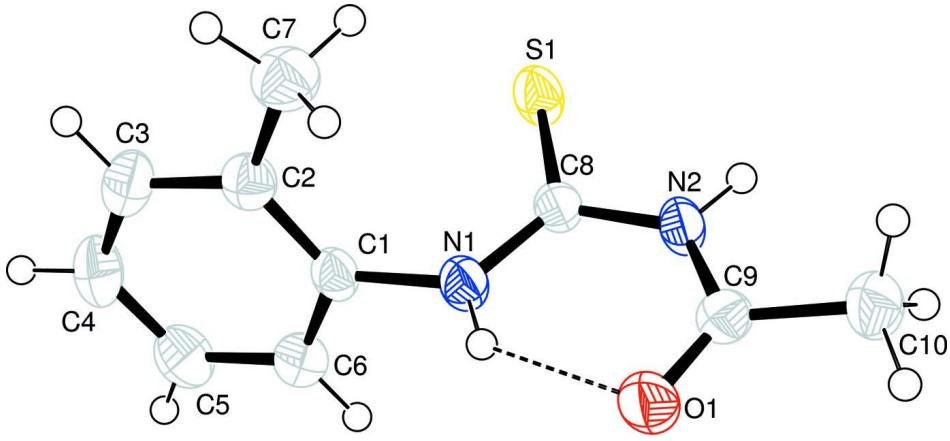

Figure 1

View of the title compound with displacement ellipsoids are drawn at the 50% probability level. The dotted lines represent the intra-molecular H-bondings.

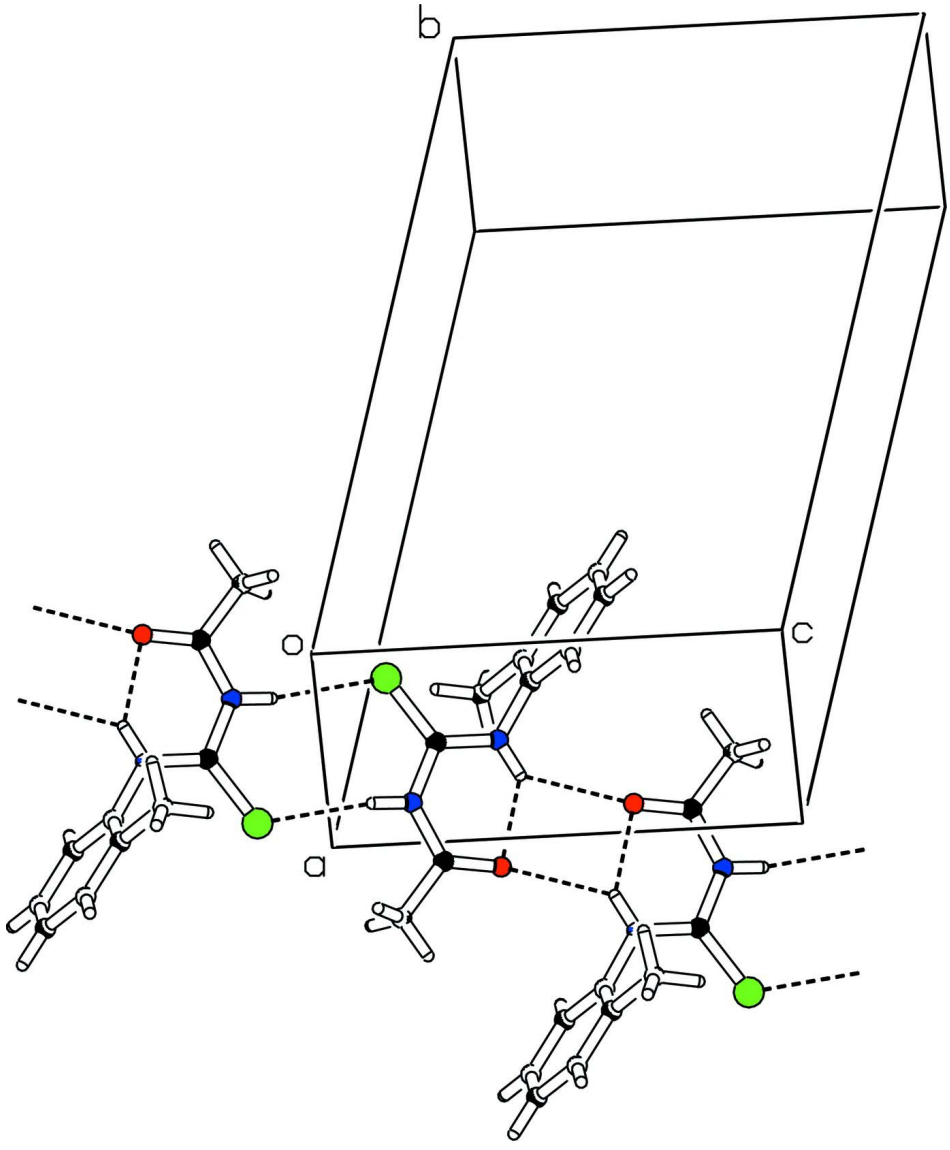

Figure 2

The partial packing (*PLATON*; Spek, 2009) which shows that molecules form polymeric chains extending along [1 0 1] direction.

3-Acetyl-1-(2-methylphenyl)thiourea

Crystal data

$C_{10}H_{12}N_2OS$
$M_r = 208.28$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 5.0444$ (2) Å
$b = 20.7019$ (9) Å
$c = 9.9464$ (4) Å
$\beta = 95.116$ (2)°
$V = 1034.55$ (7) Å^3
$Z = 4$
$F(000) = 440$
$D_x = 1.337$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1513 reflections
$\theta = 2.0$–$25.2°$
$\mu = 0.28$ mm^{-1}
$T = 296$ K
Needle, white
0.35 × 0.15 × 0.13 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.00 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.915, T_{max} = 0.938
7696 measured reflections
1812 independent reflections
1512 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 25.2°, θ_{min} = 2.0°
h = −5→6
k = −24→22
l = −11→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.088
S = 1.17
1812 reflections
129 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0311P)^2 + 0.355P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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	0.39407 (11)	0.07983 (3)	0.11730 (5)	0.0401 (2)
O1	1.0674 (3)	−0.02473 (7)	0.36353 (14)	0.0456 (5)
N1	0.7141 (3)	0.07120 (8)	0.34350 (16)	0.0337 (5)
N2	0.7701 (3)	−0.00655 (8)	0.18163 (16)	0.0326 (5)
C1	0.5997 (4)	0.12750 (10)	0.40024 (19)	0.0312 (6)
C2	0.6773 (4)	0.18879 (10)	0.3631 (2)	0.0348 (7)
C3	0.5651 (4)	0.24080 (11)	0.4260 (2)	0.0440 (8)
C4	0.3876 (4)	0.23224 (12)	0.5219 (2)	0.0493 (8)
C5	0.3150 (5)	0.17099 (12)	0.5564 (2)	0.0491 (8)
C6	0.4207 (4)	0.11826 (11)	0.4958 (2)	0.0399 (7)
C7	0.8746 (4)	0.19878 (12)	0.2608 (2)	0.0462 (8)
C8	0.6379 (4)	0.04791 (9)	0.22219 (19)	0.0294 (6)
C9	0.9799 (4)	−0.03869 (10)	0.2492 (2)	0.0334 (7)
C10	1.0907 (4)	−0.09266 (11)	0.1711 (2)	0.0456 (8)
H1	0.83956	0.05146	0.39124	0.0404*
H2	0.71344	−0.02218	0.10425	0.0391*
H3	0.61134	0.28255	0.40262	0.0528*

H4	0.31710	0.26787	0.56314	0.0591*
H5	0.19428	0.16508	0.62076	0.0589*
H6	0.37200	0.07669	0.51911	0.0478*
H7A	0.79640	0.18600	0.17331	0.0692*
H7B	1.03029	0.17317	0.28474	0.0692*
H7C	0.92326	0.24358	0.25886	0.0692*
H10A	1.21650	−0.11680	0.22928	0.0684*
H10B	1.17794	−0.07517	0.09732	0.0684*
H10C	0.94861	−0.12060	0.13674	0.0684*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0437 (3)	0.0418 (3)	0.0328 (3)	0.0132 (3)	−0.0077 (2)	−0.0084 (3)
O1	0.0543 (9)	0.0469 (10)	0.0337 (9)	0.0164 (7)	−0.0069 (7)	−0.0033 (7)
N1	0.0381 (9)	0.0333 (10)	0.0284 (9)	0.0089 (7)	−0.0038 (7)	−0.0049 (7)
N2	0.0363 (9)	0.0318 (10)	0.0289 (9)	0.0056 (7)	−0.0007 (7)	−0.0084 (7)
C1	0.0328 (10)	0.0321 (11)	0.0272 (10)	0.0042 (9)	−0.0048 (8)	−0.0054 (9)
C2	0.0321 (11)	0.0369 (12)	0.0346 (11)	−0.0007 (9)	−0.0013 (9)	−0.0017 (10)
C3	0.0483 (13)	0.0303 (12)	0.0527 (14)	−0.0017 (10)	0.0010 (11)	−0.0058 (11)
C4	0.0546 (14)	0.0419 (14)	0.0519 (15)	0.0060 (11)	0.0082 (12)	−0.0167 (12)
C5	0.0532 (14)	0.0556 (16)	0.0409 (13)	0.0045 (12)	0.0173 (11)	−0.0064 (12)
C6	0.0462 (12)	0.0376 (13)	0.0361 (12)	−0.0007 (10)	0.0057 (10)	0.0002 (10)
C7	0.0451 (13)	0.0466 (14)	0.0474 (14)	−0.0053 (11)	0.0077 (10)	0.0029 (11)
C8	0.0316 (10)	0.0289 (11)	0.0278 (10)	−0.0013 (9)	0.0041 (8)	−0.0022 (8)
C9	0.0357 (11)	0.0322 (11)	0.0326 (12)	0.0035 (9)	0.0043 (9)	0.0022 (9)
C10	0.0503 (13)	0.0436 (14)	0.0429 (13)	0.0158 (11)	0.0041 (10)	−0.0044 (11)

Geometric parameters (Å, º)

S1—C8	1.676 (2)	C4—C5	1.372 (3)
O1—C9	1.217 (2)	C5—C6	1.377 (3)
N1—C1	1.438 (3)	C9—C10	1.497 (3)
N1—C8	1.324 (2)	C3—H3	0.9300
N2—C8	1.388 (3)	C4—H4	0.9300
N2—C9	1.374 (3)	C5—H5	0.9300
N1—H1	0.8600	C6—H6	0.9300
N2—H2	0.8600	C7—H7A	0.9600
C1—C2	1.388 (3)	C7—H7B	0.9600
C1—C6	1.381 (3)	C7—H7C	0.9600
C2—C7	1.500 (3)	C10—H10A	0.9600
C2—C3	1.391 (3)	C10—H10B	0.9600
C3—C4	1.377 (3)	C10—H10C	0.9600
C1—N1—C8	123.96 (16)	O1—C9—N2	122.77 (19)
C8—N2—C9	128.34 (17)	C2—C3—H3	119.00
C1—N1—H1	118.00	C4—C3—H3	119.00
C8—N1—H1	118.00	C3—C4—H4	120.00
C8—N2—H2	116.00	C5—C4—H4	120.00
C9—N2—H2	116.00	C4—C5—H5	120.00

N1—C1—C2	120.27 (17)	C6—C5—H5	120.00
N1—C1—C6	117.88 (18)	C1—C6—H6	120.00
C2—C1—C6	121.80 (19)	C5—C6—H6	120.00
C3—C2—C7	121.32 (19)	C2—C7—H7A	109.00
C1—C2—C3	116.90 (18)	C2—C7—H7B	109.00
C1—C2—C7	121.79 (19)	C2—C7—H7C	109.00
C2—C3—C4	121.9 (2)	H7A—C7—H7B	109.00
C3—C4—C5	119.8 (2)	H7A—C7—H7C	109.00
C4—C5—C6	120.1 (2)	H7B—C7—H7C	109.00
C1—C6—C5	119.6 (2)	C9—C10—H10A	109.00
S1—C8—N1	124.12 (15)	C9—C10—H10B	109.00
S1—C8—N2	118.97 (14)	C9—C10—H10C	109.00
N1—C8—N2	116.91 (17)	H10A—C10—H10B	109.00
O1—C9—C10	122.74 (18)	H10A—C10—H10C	109.00
N2—C9—C10	114.49 (17)	H10B—C10—H10C	110.00

C8—N1—C1—C2	−79.9 (3)	C6—C1—C2—C3	−0.4 (3)
C8—N1—C1—C6	102.7 (2)	C6—C1—C2—C7	179.11 (19)
C1—N1—C8—S1	−0.9 (3)	N1—C1—C6—C5	177.42 (18)
C1—N1—C8—N2	179.42 (17)	C2—C1—C6—C5	0.1 (3)
C9—N2—C8—S1	177.45 (16)	C1—C2—C3—C4	0.8 (3)
C9—N2—C8—N1	−2.8 (3)	C7—C2—C3—C4	−178.76 (19)
C8—N2—C9—O1	5.5 (3)	C2—C3—C4—C5	−0.8 (3)
C8—N2—C9—C10	−175.01 (18)	C3—C4—C5—C6	0.4 (3)
N1—C1—C2—C3	−177.70 (17)	C4—C5—C6—C1	−0.1 (3)
N1—C1—C2—C7	1.8 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1	0.86	1.99	2.664 (2)	135
N1—H1···O1^{i}	0.86	2.50	3.172 (2)	135
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