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3-Acetyl-1-(3-chlorophenyl)thiourea

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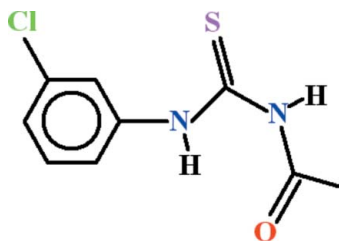
Received 18 March 2012; accepted 21 March 2012

 Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.034; wR factor = 0.109; data-to-parameter ratio = 13.9.

In the title compound, $\text{C}_9\text{H}_9\text{ClN}_2\text{OS}$, the 3-chlorophenyl and acetylthiourea fragments are oriented at a dihedral angle of $62.68(5)^\circ$. An intramolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bond generates an $S(6)$ ring motif. Molecules are linked into dimers via a cyclic $R_2^2(8)$ motif of $\text{N}-\text{H}\cdots\text{S}$ hydrogen bonds. These dimers are further connected through $\text{C}-\text{H}\cdots\text{S}$ interactions, completing an $R_2^2(12)$ motif, into chains along $[010]$.

Related literature

For related structures, see: Shahwar *et al.* (2012*a,b*); For graph-set notation, see: Bernstein *et al.* (1995).



Experimental

Crystal data

 $\text{C}_9\text{H}_9\text{ClN}_2\text{OS}$
 $M_r = 228.69$

 Monoclinic, $C2/c$
 $a = 28.3980(14)$ Å

 $b = 4.1768(2)$ Å

 $c = 20.2635(11)$ Å

 $\beta = 122.651(2)^\circ$
 $V = 2023.69(18)$ Å³
 $Z = 8$

 Mo $K\alpha$ radiation

 $\mu = 0.55$ mm⁻¹
 $T = 296$ K

 $0.35 \times 0.22 \times 0.22$ mm

Data collection

Bruker Kappa APEXII CCD diffractometer

 Absorption correction: multi-scan (*SADABS*; Bruker, 2005)

 $T_{\min} = 0.868$, $T_{\max} = 0.872$

7045 measured reflections

1775 independent reflections

 1450 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.029$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.034$
 $wR(F^2) = 0.109$
 $S = 1.11$

1775 reflections

128 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.23$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.23$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1}\cdots\text{O1}$	0.86	1.96	2.648 (3)	136
$\text{N2}-\text{H2}\cdots\text{S1}^i$	0.86	2.56	3.4095 (18)	170
$\text{C9}-\text{H9A}\cdots\text{S1}^{ii}$	0.96	2.85	3.799 (3)	170

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

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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2470).

References

- Bernstein, J., Davis, R. E., Shimon, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
- Bruker (2005). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (2009). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Shahwar, D., Tahir, M. N., Chohan, M. M., Ahmad, N. & Raza, M. A. (2012*a*). *Acta Cryst.*, **E68**, o1160.
- Shahwar, D., Tahir, M. N., Chohan, M. M., Ahmad, N. & Samiullah, (2012*b*). *Acta Cryst.* **E68**, o508.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.

supporting information

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3-Acetyl-1-(3-chlorophenyl)thiourea

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S1. Comment

The title compound (Fig. 1) has been synthesized as a continuation of our work to find new enzyme inhibitors.

The crystal structures of *N*-(2-methylphenylcarbamothioyl)acetamide (Shahwar *et al.*, 2012*a*) and *N*-(phenylcarbamothioyl)acetamide (Shahwar *et al.*, 2012*b*) have been reported which are related to the title compound.

In the title compound, the 3-chlorophenyl group A (C1–C6/C11) and the *N*-carbamothioylacetamide moiety B (N1/C7/S1/N2/C8/O1/C9) are planar with r. m. s. deviation of 0.0055 Å and 0.0060 Å, respectively. The dihedral angle between A/B is 62.68 (5)°. There exist 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*₂²(8) ring motifs (Table 1, Fig. 2). The dimers are interlinked due to C—H···S H-bondings (Table 1, Fig. 2) and complete *R*₂²(12) ring motifs.

S2. Experimental

The title compound 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 3-chloroaniline (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 from ethyl acetate yielded colorless rods (m.p. 374 K).

S3. Refinement

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

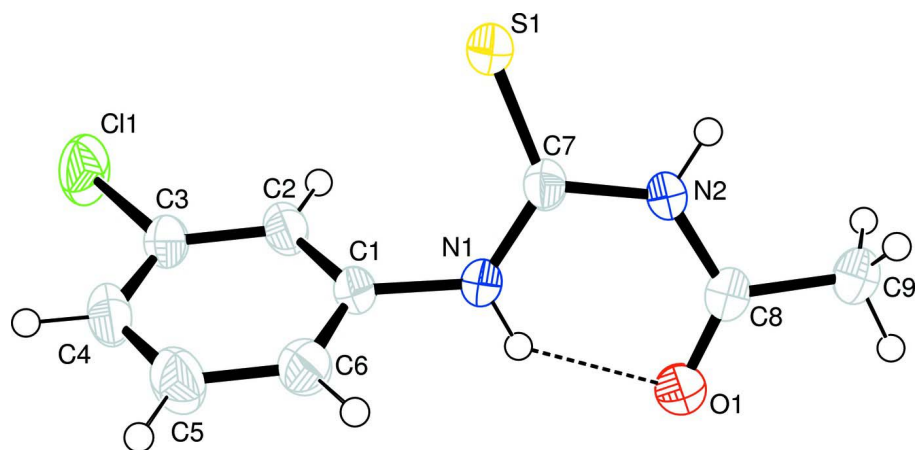


Figure 1

View of the title compound with displacement ellipsoids drawn at the 50% probability level. The dotted lines represent the intramolecular hydrogen bond.

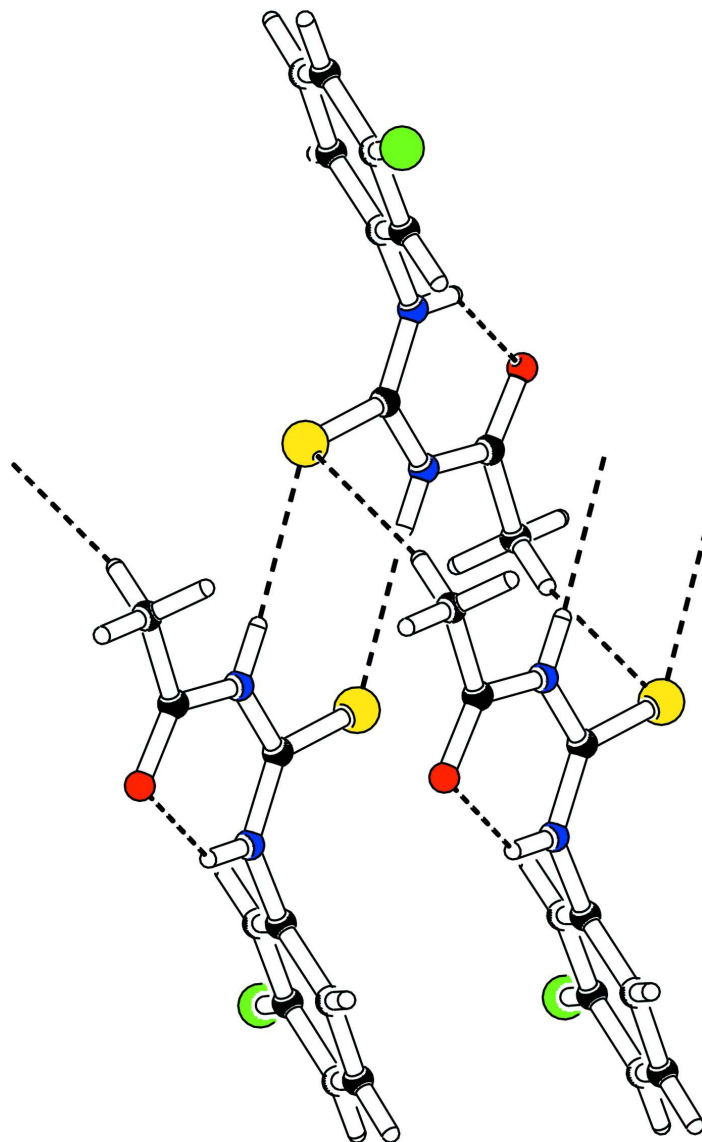


Figure 2

The partial packing (*PLATON*; Spek, 2009) showing molecules connected *via* N-H \cdots O and C-H \cdots S interactions.

3-Acetyl-1-(3-chlorophenyl)thiourea

Crystal data

$C_9H_9ClN_2OS$

$M_r = 228.69$

Monoclinic, $C2/c$

Hall symbol: $-C 2yc$

$a = 28.3980$ (14) Å

$b = 4.1768$ (2) Å

$c = 20.2635$ (11) Å

$\beta = 122.651$ (2)°

$V = 2023.69$ (18) Å³

$Z = 8$

$F(000) = 944$

$D_x = 1.501$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1464 reflections

$\theta = 2.1$ – 25.1 °

$\mu = 0.55$ mm⁻¹

$T = 296$ K

Rod, colorless

$0.35 \times 0.22 \times 0.22$ mm

Data collection

Bruker Kappa APEXII CCD
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 Detector resolution: 8.10 pixels mm⁻¹
 ω scans
 Absorption correction: multi-scan
 (SADABS; Bruker, 2005)
 $T_{\min} = 0.868$, $T_{\max} = 0.872$

7045 measured reflections
 1745 independent reflections
 1450 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.029$
 $\theta_{\max} = 25.1^\circ$, $\theta_{\min} = 2.1^\circ$
 $h = -33 \rightarrow 27$
 $k = -4 \rightarrow 3$
 $l = -19 \rightarrow 24$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.034$
 $wR(F^2) = 0.109$
 $S = 1.11$
 1775 reflections
 128 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
 $w = 1/[\sigma^2(F_o^2) + (0.0527P)^2 + 1.9073P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.23 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.23 \text{ e } \text{\AA}^{-3}$

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.27943 (3)	0.9289 (2)	0.34233 (4)	0.0593 (3)
S1	0.08011 (2)	0.46913 (17)	0.10724 (3)	0.0436 (2)
O1	-0.03091 (7)	1.0496 (5)	0.16023 (11)	0.0610 (7)
N1	0.06728 (7)	0.7792 (5)	0.21108 (10)	0.0383 (6)
N2	-0.00930 (7)	0.7704 (5)	0.08375 (10)	0.0353 (6)
C1	0.12315 (9)	0.7158 (6)	0.27507 (12)	0.0334 (7)
C2	0.16843 (9)	0.8388 (6)	0.27522 (13)	0.0375 (8)
C3	0.22127 (9)	0.7785 (6)	0.33950 (13)	0.0375 (7)
C4	0.22942 (10)	0.6054 (7)	0.40248 (14)	0.0461 (8)
C5	0.18373 (11)	0.4900 (7)	0.40138 (16)	0.0520 (9)
C6	0.13025 (10)	0.5409 (6)	0.33725 (14)	0.0422 (8)
C7	0.04606 (9)	0.6845 (5)	0.13794 (12)	0.0328 (7)
C8	-0.04474 (9)	0.9441 (6)	0.09596 (14)	0.0395 (8)
C9	-0.10196 (10)	0.9910 (7)	0.02487 (15)	0.0480 (9)
H1	0.04591	0.88682	0.22087	0.0459*
H2	-0.02292	0.70566	0.03650	0.0424*
H3	0.16340	0.95861	0.23319	0.0450*

H4	0.26537	0.56692	0.44526	0.0553*
H5	0.18883	0.37642	0.44424	0.0624*
H6	0.09947	0.45780	0.33622	0.0506*
H9A	-0.09942	1.10785	-0.01390	0.0720*
H9B	-0.12438	1.10907	0.03877	0.0720*
H9C	-0.11887	0.78623	0.00402	0.0720*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0285 (4)	0.0836 (6)	0.0561 (5)	-0.0088 (3)	0.0164 (3)	0.0058 (4)
S1	0.0301 (4)	0.0600 (4)	0.0331 (4)	0.0091 (3)	0.0120 (3)	-0.0070 (3)
O1	0.0378 (11)	0.0981 (16)	0.0396 (11)	0.0147 (10)	0.0159 (9)	-0.0173 (10)
N1	0.0223 (10)	0.0590 (13)	0.0290 (10)	0.0031 (9)	0.0109 (8)	-0.0061 (9)
N2	0.0231 (9)	0.0509 (12)	0.0251 (10)	0.0023 (8)	0.0086 (8)	-0.0049 (8)
C1	0.0248 (11)	0.0434 (13)	0.0262 (12)	0.0014 (9)	0.0099 (9)	-0.0051 (9)
C2	0.0303 (13)	0.0479 (14)	0.0283 (12)	-0.0001 (10)	0.0119 (10)	0.0032 (10)
C3	0.0250 (12)	0.0480 (14)	0.0334 (12)	-0.0038 (10)	0.0117 (10)	-0.0051 (10)
C4	0.0302 (13)	0.0605 (16)	0.0342 (14)	0.0060 (11)	0.0085 (11)	0.0070 (12)
C5	0.0439 (16)	0.0684 (18)	0.0374 (14)	0.0033 (13)	0.0178 (13)	0.0173 (13)
C6	0.0340 (14)	0.0545 (15)	0.0383 (14)	-0.0041 (11)	0.0196 (11)	0.0004 (11)
C7	0.0242 (12)	0.0400 (13)	0.0298 (12)	-0.0014 (9)	0.0116 (10)	-0.0003 (10)
C8	0.0274 (13)	0.0504 (15)	0.0371 (14)	0.0019 (10)	0.0150 (11)	-0.0017 (11)
C9	0.0304 (14)	0.0653 (17)	0.0412 (14)	0.0124 (12)	0.0146 (12)	-0.0002 (12)

Geometric parameters (Å, °)

Cl1—C3	1.739 (3)	C3—C4	1.373 (4)
S1—C7	1.667 (3)	C4—C5	1.373 (5)
O1—C8	1.222 (3)	C5—C6	1.382 (4)
N1—C1	1.431 (3)	C8—C9	1.493 (4)
N1—C7	1.324 (3)	C2—H3	0.9300
N2—C7	1.394 (3)	C4—H4	0.9300
N2—C8	1.367 (4)	C5—H5	0.9300
N1—H1	0.8600	C6—H6	0.9300
N2—H2	0.8600	C9—H9A	0.9600
C1—C6	1.374 (3)	C9—H9B	0.9600
C1—C2	1.383 (4)	C9—H9C	0.9600
C2—C3	1.379 (4)		
C1—N1—C7	124.9 (2)	N1—C7—N2	116.3 (2)
C7—N2—C8	128.42 (19)	O1—C8—C9	122.5 (3)
C1—N1—H1	118.00	N2—C8—C9	114.8 (2)
C7—N1—H1	118.00	O1—C8—N2	122.7 (2)
C7—N2—H2	116.00	C1—C2—H3	121.00
C8—N2—H2	116.00	C3—C2—H3	121.00
N1—C1—C2	120.5 (2)	C3—C4—H4	120.00
N1—C1—C6	118.1 (3)	C5—C4—H4	120.00

C2—C1—C6	121.3 (2)	C4—C5—H5	120.00
C1—C2—C3	118.1 (2)	C6—C5—H5	120.00
C11—C3—C4	118.6 (2)	C1—C6—H6	120.00
C2—C3—C4	121.7 (3)	C5—C6—H6	120.00
C11—C3—C2	119.68 (19)	C8—C9—H9A	109.00
C3—C4—C5	119.1 (3)	C8—C9—H9B	109.00
C4—C5—C6	120.7 (3)	C8—C9—H9C	109.00
C1—C6—C5	119.2 (3)	H9A—C9—H9B	109.00
S1—C7—N1	125.0 (2)	H9A—C9—H9C	109.00
S1—C7—N2	118.65 (16)	H9B—C9—H9C	109.00
C7—N1—C1—C2	63.5 (3)	C6—C1—C2—C3	0.6 (4)
C7—N1—C1—C6	-119.0 (3)	N1—C1—C6—C5	-176.9 (2)
C1—N1—C7—S1	1.1 (4)	C2—C1—C6—C5	0.6 (4)
C1—N1—C7—N2	-179.9 (2)	C1—C2—C3—C11	-179.9 (2)
C8—N2—C7—S1	179.9 (2)	C1—C2—C3—C4	-0.8 (4)
C8—N2—C7—N1	0.8 (4)	C11—C3—C4—C5	179.0 (2)
C7—N2—C8—O1	-0.3 (4)	C2—C3—C4—C5	-0.1 (4)
C7—N2—C8—C9	-179.5 (2)	C3—C4—C5—C6	1.3 (4)
N1—C1—C2—C3	178.0 (2)	C4—C5—C6—C1	-1.6 (4)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1...O1	0.86	1.96	2.648 (3)	136
N2—H2...S1 ⁱ	0.86	2.56	3.4095 (18)	170
C9—H9A...S1 ⁱⁱ	0.96	2.85	3.799 (3)	170

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