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

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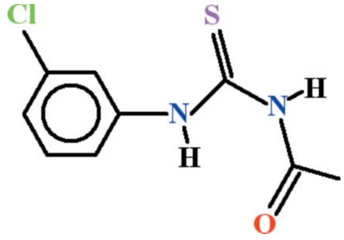
Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.034; $wR$ factor = 0.109; data-to-parameter ratio = 13.9.

In the title compound, $C_9H_9ClN_2OS$, the 3-chlorophenyl and acetylthiourea fragments are oriented at a dihedral angle of 62.68 (5)°. An intramolecular N—H···O hydrogen bond generates an $S(6)$ ring motif. Molecules are linked into dimers *via* a cyclic $R_2^2(8)$ motif of N—H···S hydrogen bonds. These dimers are further connected through C—H···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*

| | |
|---|---|
| $C_9H_9ClN_2OS$ | $V$ = 2023.69 (18) Å$^3$ |
| $M_r$ = 228.69 | $Z$ = 8 |
| Monoclinic, $C2/c$ | Mo $K\alpha$ radiation |
| $a$ = 28.3980 (14) Å | $\mu$ = 0.55 mm$^{-1}$ |
| $b$ = 4.1768 (2) Å | $T$ = 296 K |
| $c$ = 20.2635 (11) Å | 0.35 × 0.22 × 0.22 mm |
| $\beta$ = 122.651 (2)° | |

### *Data collection*

| | |
|---|---|
| Bruker Kappa APEXII CCD diffractometer | 7045 measured reflections |
| Absorption correction: multi-scan (*SADABS*; Bruker, 2005) | 1775 independent reflections |
| $T_{min}$ = 0.868, $T_{max}$ = 0.872 | 1450 reflections with $I > 2\sigma(I)$ |
| | $R_{int}$ = 0.029 |

### *Refinement*

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)]$ = 0.034 | 128 parameters |
| $wR(F^2)$ = 0.109 | H-atom parameters constrained |
| $S$ = 1.11 | $\Delta\rho_{max}$ = 0.23 e Å$^{-3}$ |
| 1775 reflections | $\Delta\rho_{min}$ = −0.23 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 | 1.96 | 2.648 (3) | 136 |
| N2—H2···S1$^{i}$ | 0.86 | 2.56 | 3.4095 (18) | 170 |
| C9—H9$A$···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*.

The authors acknowledge the provision of funds for the purchase of the diffractometer and encouragement by Dr Muhammad Akram Chaudhary, Vice Chancellor, University of Sargodha, Pakistan. The authors 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: GK2470).

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# supplementary materials

*Acta Cryst.* (2012). E**68**, o1189 [doi:10.1107/S1600536812012147]

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

**Durre Shahwar, M. Nawaz Tahir, Muhammad Mansha Chohan, Naeem Ahmad and M. Asam Raza**

### 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/Cl1) 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 $R_2^2(8)$ ring motifs (Table 1, Fig. 2). The dimers are interlinked due to C—H···S H–bondings (Table 1, Fig. 2) and complete $R_2^2(12)$ ring motifs.

### 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).

### 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).

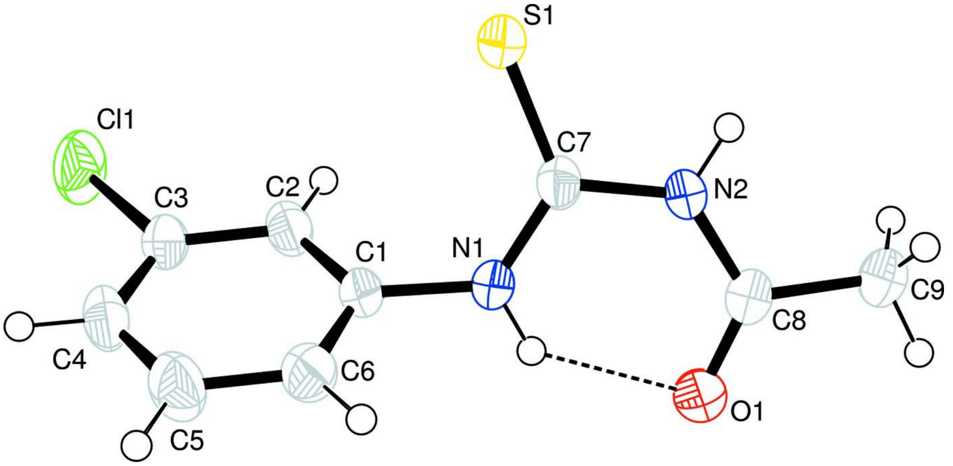

**Figure 1**

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

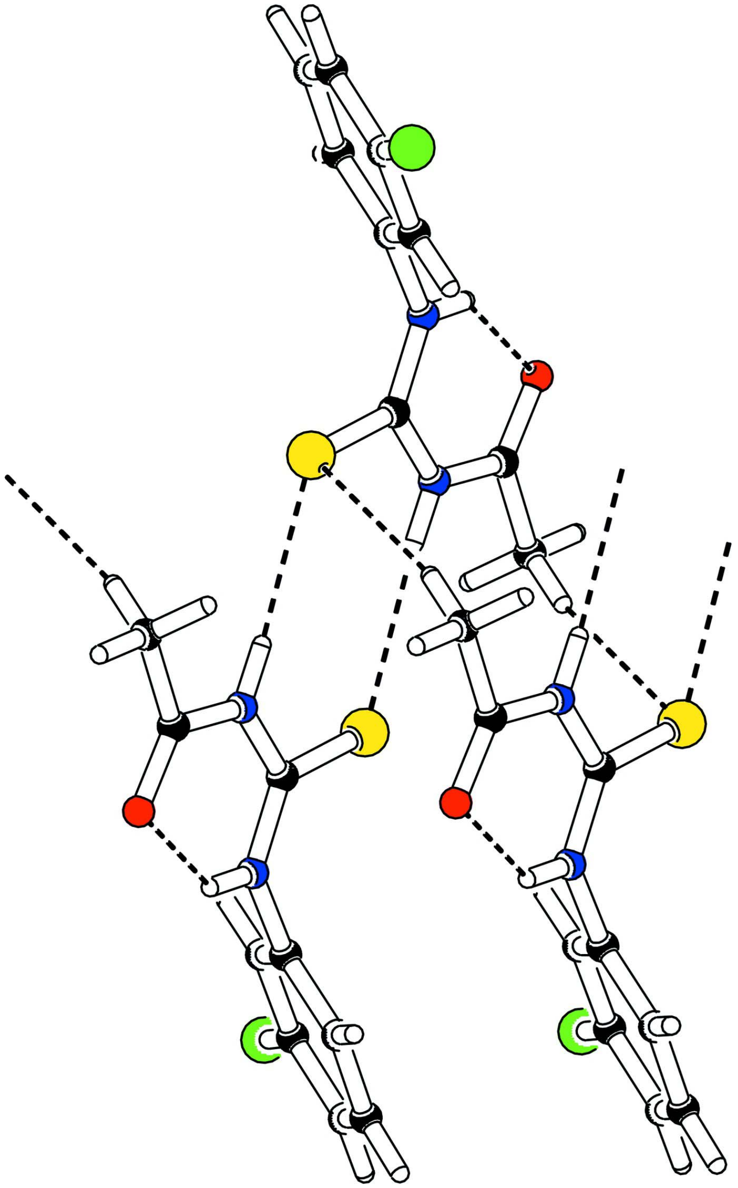

**Figure 2**

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

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

*Crystal data*

| | |
|---|---|
| $C_9H_9ClN_2OS$ | $F(000) = 944$ |
| $M_r = 228.69$ | $D_x = 1.501$ Mg m$^{-3}$ |
| Monoclinic, $C2/c$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Hall symbol: -C 2yc | Cell parameters from 1464 reflections |
| $a = 28.3980$ (14) Å | $\theta = 2.1$–$25.1°$ |
| $b = 4.1768$ (2) Å | $\mu = 0.55$ mm$^{-1}$ |
| $c = 20.2635$ (11) Å | $T = 296$ K |
| $\beta = 122.651$ (2)° | Rod, colorless |
| $V = 2023.69$ (18) Å$^3$ | 0.35 × 0.22 × 0.22 mm |
| $Z = 8$ | |

*Data collection*

| | |
|---|---|
| Bruker Kappa APEXII CCD diffractometer | 7045 measured reflections |
| Radiation source: fine-focus sealed tube | 1745 independent reflections |
| Graphite monochromator | 1450 reflections with $I > 2\sigma(I)$ |
| Detector resolution: 8.10 pixels mm$^{-1}$ | $R_{\text{int}} = 0.029$ |
| $\omega$ scans | $\theta_{\text{max}} = 25.1°$, $\theta_{\text{min}} = 2.1°$ |
| Absorption correction: multi-scan (*SADABS*; Bruker, 2005) | $h = -33 \rightarrow 27$ |
| $T_{\text{min}} = 0.868$, $T_{\text{max}} = 0.872$ | $k = -4 \rightarrow 3$ |
| | $l = -19 \rightarrow 24$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.034$ | H-atom parameters constrained |
| $wR(F^2) = 0.109$ | $w = 1/[\sigma^2(F_o^2) + (0.0527P)^2 + 1.9073P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.11$ | $(\Delta/\sigma)_{\text{max}} < 0.001$ |
| 1775 reflections | $\Delta\rho_{\text{max}} = 0.23$ e Å$^{-3}$ |
| 128 parameters | $\Delta\rho_{\text{min}} = -0.23$ e Å$^{-3}$ |
| 0 restraints | |
| Primary atom site location: structure-invariant direct methods | |

*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* | $U_{\text{iso}}*/U_{\text{eq}}$ |
|---|---|---|---|---|
| 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 (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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, º)*

| | | | |
|---|---|---|---|
| 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 |

| | | | |
|---|---|---|---|
| Cl1—C3—C4 | 118.6 (2) | C1—C6—H6 | 120.00 |
| C2—C3—C4 | 121.7 (3) | C5—C6—H6 | 120.00 |
| Cl1—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—Cl1 | −179.9 (2) |
| C8—N2—C7—S1 | 179.9 (2) | C1—C2—C3—C4 | −0.8 (4) |
| C8—N2—C7—N1 | 0.8 (4) | Cl1—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, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···O1 | 0.86 | 1.96 | 2.648 (3) | 136 |
| N2—H2···S1$^{i}$ | 0.86 | 2.56 | 3.4095 (18) | 170 |
| C9—H9*A*···S1$^{ii}$ | 0.96 | 2.85 | 3.799 (3) | 170 |

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