

Synthesis, structure and computational study of 1-acetyl-3-(3,5-dichlorophenyl)thiourea

Sharatha Kumar^{a*} and Tuncer Hökelek^b

^aDepartment of Chemistry, Yenepoya Institute of Arts, Science, Commerce and Management, Mangaluru, Yenepoya University (Deemed to be university), 575013 Karnataka, India, and ^bHacettepe University, Department of Physics, 06800 Beytepe-Ankara, Türkiye. *Correspondence e-mail: sharathiascm@yenepoya.edu.in

Received 31 August 2026

Accepted 29 September 2026

Edited by M. Weil, Vienna University of Technology, Austria

Keywords: crystal structure; acetyl; thiourea; 3,5-dichlorophenyl; disorder.

CCDC reference: 1816809

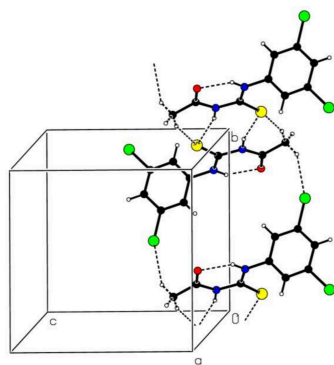
Supporting information: this article has supporting information at journals.iucr.org/e

The title compound, $C_9H_8Cl_2N_2OS$, consists of dichlorophenyl and acetyl moieties attached to the N atoms of the bridging thiourea group. The dihedral angle between the acetyl group and the thiourea group is $5.6(6)^\circ$, and they are oriented with respect to the phenyl ring at dihedral angles of $70.3(4)$ and $69.1(1)^\circ$, respectively. The molecular conformation features an intramolecular $N-H\cdots O$ hydrogen bond with an $S(6)$ ring motif. In the crystal, $C-H\cdots Cl$ and double-acceptor $N-H\cdots S$ and $C-H\cdots S$ hydrogen bonds link the molecules, enclosing $R_2^1(6)$, $R_2^2(8)$ and $R_2^2(20)$ ring motifs, into infinite chains running parallel to the b axis. Neither $\pi\cdots\pi$ nor $C-H\cdots\pi$ (ring) interactions are observed. Hirshfeld surface analysis revealed that the most important contributions for crystal packing are $H\cdots H$ (28.6%), $H\cdots Cl/Cl\cdots H$ (22.9%), $H\cdots S/S\cdots H$ (10.4%), $H\cdots O/O\cdots H$ (9.6%) and $H\cdots C/C\cdots H$ (9.0%) interactions. The volume of the crystal voids and the percentage of free space were calculated to be $62.7\text{ \AA}^3$ and 10.9%. $N-H\cdots S$, $C-H\cdots S$ and $C-H\cdots Cl$ hydrogen-bonding energies of -10.0 , -3.9 and -7.7 kJ mol^{-1} , respectively, were computed. Evaluations of the electrostatic, dispersion and total energy frameworks indicate that the crystal consolidation is dominated by dispersion energy contributions.

1. Chemical context

Acylthiourea derivatives exhibit a broad range of biological activities, including anticancer, antimicrobial, anti-inflammatory, anticonvulsant, antidiabetic and urease-inhibitory effects (Bielenica *et al.*, 2016; Ashgar *et al.*, 2018; Khan *et al.*, 2014; Canatar *et al.*, 2023). In particular, thiourea-based compounds have been investigated as anticancer agents because the thiourea pharmacophore provides hydrogen-bonding sites that can facilitate interactions with biological targets, while variations of the N-substituents allow modulation of their molecular and electronic properties (Mishra & Batra, 2013; Nammalwar *et al.*, 2013). N-substituted acylthioureas are also useful synthetic intermediates for the preparation of heterocyclic compounds and have been investigated as anion receptors, organocatalysts and corrosion inhibitors (Aly *et al.*, 2016; Bregovic & Basaric, 2015; Guang *et al.*, 2015; Al-Abbasi *et al.*, 2023). Furthermore, the presence of sulfur and carbonyl oxygen donor atoms gives the compounds versatile metal-coordination properties (Saeed *et al.*, 2013, 2017).

The structural versatility of acylthioureas is reflected in their molecular conformations and supramolecular assemblies in the solid state. Crystallographic studies have shown that acylthiourea derivatives can adopt conformations consolidated by intramolecular $N-H\cdots S$ and $N-H\cdots O$ hydrogen bonds, while intermolecular hydrogen bonds contribute significantly to the formation of extended supramolecular networks (Gowda *et al.*, 2012b; Kumar *et al.*, 2012a; Yusof *et*

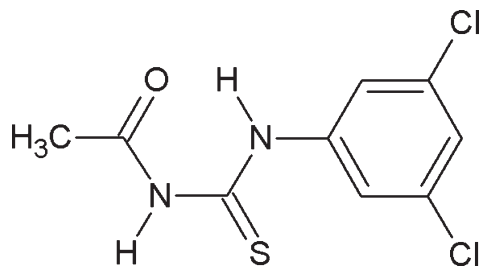


OPEN ACCESS

Published under a CC BY 4.0 licence

al., 2012). The molecular conformation and hydrogen-bonding patterns are influenced by the nature of substituents and by the conjugated acylthiourea frameworks (Saeed *et al.*, 2014).

As part of our studies in this area, we now describe the synthesis and structure of the title compound, $C_9H_8Cl_2N_2OS$, (**I**).



2. Structural commentary

Compound (**I**) consists of dichlorophenyl and acetyl moieties bridged by a thiourea group (Fig. 1). The two N—H bonds, like the thiocarbonyl C=S and carbonyl C=O bonds, are in pseudo-*anti* conformations. The acetyl (O1/C8/C9) group is oriented with respect to the statistically planar thiourea (S1/C7/N1/N2) group [root-mean-square value of 0.004 (4) Å] at a dihedral angle of 5.6 (6)°; the dihedral angles between the phenyl ring (C1–C6) and acetyl and thiourea groups are 70.3 (4) and 69.1 (1)°, respectively. The Cl1, Cl2 and N1 atoms are −0.037 (2), −0.016 (2) and −0.034 (4) Å, respectively, away from the least-squares plane of the phenyl ring. All bond lengths are in normal ranges (Allen *et al.*, 1987) and comparable to those in the similar compounds *N*-(2-chlorophenyl)-*N'*-propanoyl-thiourea (**II**) (Kumar & Hökelek, 2026a) and *N*-acetyl-*N'*-(4-chlorophenyl) thiourea (**III**) (Kumar & Hökelek, 2026b). An intramolecular N—H...O hydrogen bond (Table 1) forms an S(6) ring motif (Etter *et al.*, 1990) and stabilizes the molecular conformation (Fig. 1).

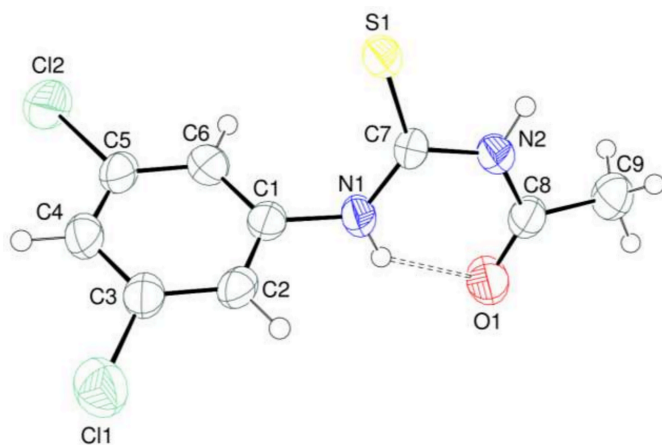


Figure 1

The asymmetric unit of (**I**) with displacement ellipsoids drawn at the 50% probability level. The intramolecular N—H...O hydrogen bond is shown as a dashed line. Only the major components of the disordered methyl H atoms are shown for clarity.

Table 1

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—H1N...O1	0.86 (2)	1.95 (4)	2.655 (5)	138 (5)
N2—H2N...S1 ⁱ	0.86 (2)	2.57 (2)	3.395 (4)	163 (5)
C9—H9F...S1 ⁱ	0.96	2.66	3.576 (5)	160
C9—H9E...Cl1 ⁱⁱ	0.96	2.62	3.478 (6)	149

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

The C1—N1—C7 [123.9 (4)°], S1—C7—N1 [124.4 (3)°] and N2—C8—C9 [113.6 (4)°] bond angles in (**I**) are significantly narrowed when compared with the corresponding values in compounds (**II**) and (**III**). Comparison with other structures is provided in the Database survey.

3. Supramolecular features

In the crystal, C—H...Cl and double-acceptor N—H...S and C—H...S hydrogen bonds (Table 1) link the molecules into infinite chains extending parallel to the *b* axis, enclosing $R_2^1(6)$, $R_2^2(8)$ and $R_2^2(20)$ ring motifs (Etter *et al.*, 1990; Fig. 2). Significant π ... π or C—H... π (ring) interactions are not observed.

The intermolecular interactions in the crystal were quantified and visualized by carrying out a Hirshfeld surface (HS) analysis using *CrystalExplorer* (Spackman *et al.*, 2021). It is noted that only the major components of the disordered part

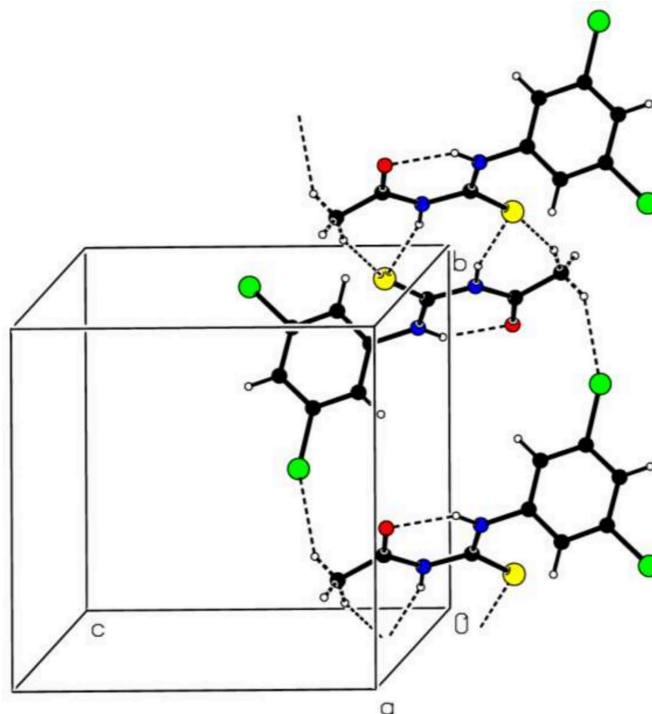


Figure 2

A partial packing diagram showing the infinite hydrogen-bonded chains extending parallel to the *b* axis. The intramolecular N—H...O and intermolecular N—H...S, C—H...S and C—H...Cl hydrogen bonds are shown as dashed lines with the $S(6)$, $R_2^1(6)$, $R_2^2(8)$ and $R_2^2(20)$ ring motifs. Only the hydrogen-bonded components of the disordered atoms are shown for clarity.

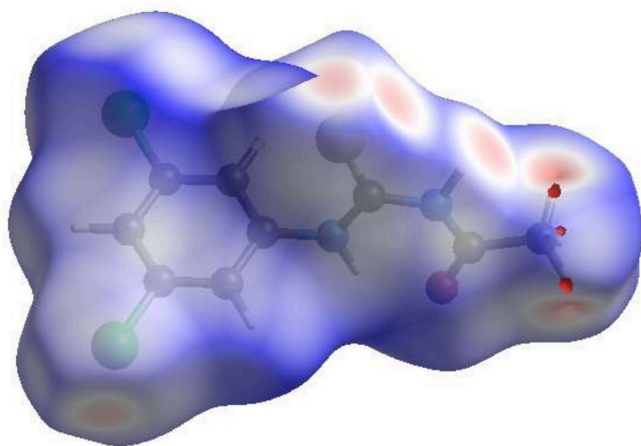


Figure 3
View of the three-dimensional Hirshfeld surface of (**I**) plotted over d_{norm} .

of the methyl group (C9) were taken into account for the analysis. Fig. 3 shows the HS plotted over d_{norm} in the crystal. The white surface indicates contacts with distances equal to the sum of van der Waals radii, and the red and blue colours indicate distances shorter (in close contact) or longer (distinct contacts) than the van der Waals radii, respectively. The red spots indicate their roles as the respective donors and/or acceptors atoms in hydrogen-bonding, as discussed. They also appear as the blue and red regions corresponding to positive and negative potentials on the HS mapped over electrostatic potential, as shown in Fig. 4. The blue and red regions indicate positive (hydrogen-bond donors) and negative (hydrogen-bond acceptors) electrostatic potentials.

The overall two-dimensional fingerprint plot is shown in Fig. 5a and delineated into different contact types in Fig. 5b–r. According to the two-dimensional fingerprint plots, the $\text{H}\cdots\text{H}$, $\text{H}\cdots\text{Cl}/\text{Cl}\cdots\text{H}$, $\text{H}\cdots\text{S}/\text{S}\cdots\text{H}$, $\text{H}\cdots\text{O}/\text{O}\cdots\text{H}$ and $\text{H}\cdots\text{C}/\text{C}\cdots\text{H}$ contacts make the most significant contributions to the HS, at 28.6%, 22.9%, 10.4%, 9.6% and 9.0%, respectively.

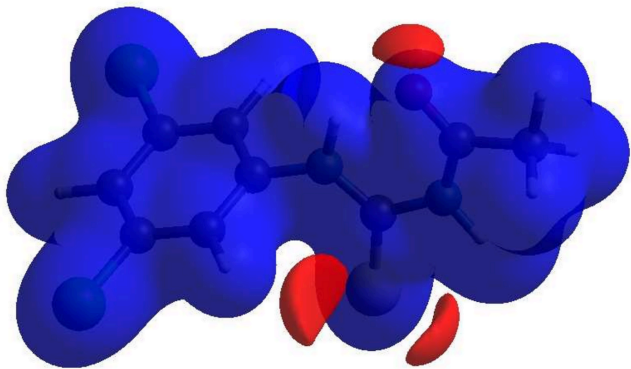


Figure 4
View of the three-dimensional Hirshfeld surface of the title compound plotted over electrostatic potential. Hydrogen-bonding donors and acceptors are shown as blue and red regions around the atoms, corresponding to positive and negative potentials, respectively.

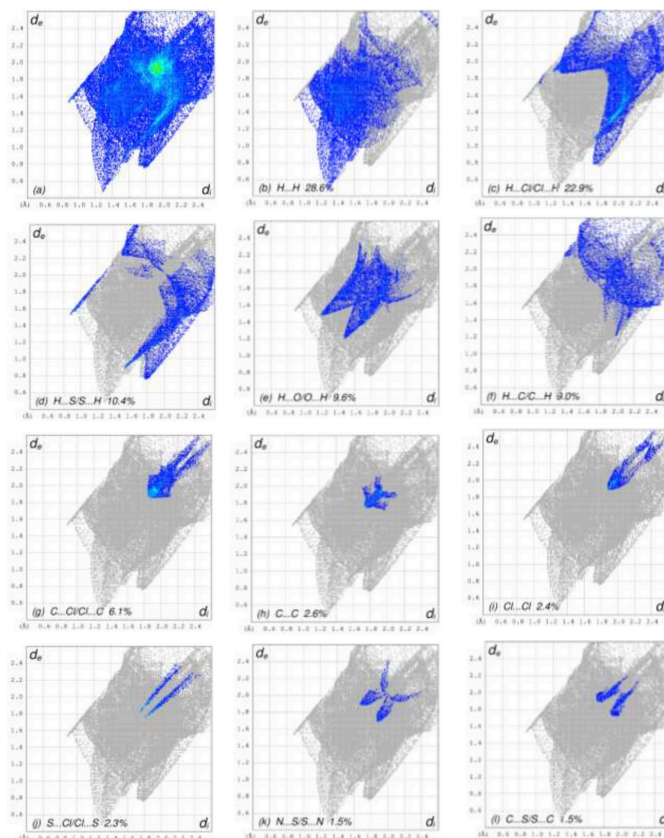


Figure 5

The full two-dimensional fingerprint plots for (**I**), showing (a) all interactions, and delineated into (b) $\text{H}\cdots\text{H}$, (c) $\text{H}\cdots\text{Cl}/\text{Cl}\cdots\text{H}$, (d) $\text{H}\cdots\text{S}/\text{S}\cdots\text{H}$, (e) $\text{H}\cdots\text{O}/\text{O}\cdots\text{H}$, (f) $\text{H}\cdots\text{C}/\text{C}\cdots\text{H}$, (g) $\text{C}\cdots\text{Cl}/\text{Cl}\cdots\text{C}$, (h) $\text{C}\cdots\text{C}$, (i) $\text{Cl}\cdots\text{Cl}$, (j) $\text{S}\cdots\text{Cl}/\text{Cl}\cdots\text{S}$, (k) $\text{N}\cdots\text{S}/\text{S}\cdots\text{N}$, (l) $\text{C}\cdots\text{S}/\text{S}\cdots\text{C}$, (m) $\text{C}\cdots\text{O}/\text{O}\cdots\text{C}$, (n) $\text{H}\cdots\text{N}/\text{N}\cdots\text{H}$, (o) $\text{N}\cdots\text{O}/\text{O}\cdots\text{N}$, (p) $\text{O}\cdots\text{Cl}/\text{Cl}\cdots\text{O}$ and (r) $\text{O}\cdots\text{S}/\text{S}\cdots\text{O}$ interactions. The d_i and d_e values are the closest internal and external distances (in Å) from given points on the Hirshfeld surface.

The volume of the crystal voids (Fig. 6a,b) and the percentage of free space in the unit cell was calculated as 62.70 Å^3 and 10.86%, respectively, showing that there are no large cavities in the crystal packing.

The intermolecular interaction energies were computed on basis of the CE-B3LYP/6-31G(d,p) energy model available in *CrystalExplorer* (Spackman *et al.*, 2021), where a cluster of molecules is generated by applying crystallographic symmetry operations with respect to a selected central molecule within the radius of 3.8 Å by default. The total intermolecular energy (E_{tot}) is the sum of electrostatic (E_{ele}), polarization (E_{pol}), dispersion (E_{dis}) and exchange-repulsion (E_{rep}) energies

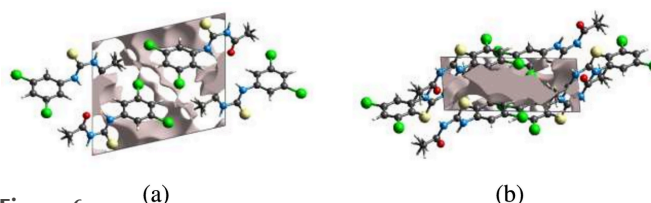


Figure 6
Crystal voids in the packing of (**I**) viewed down the (a) a axis and (b) b axis.

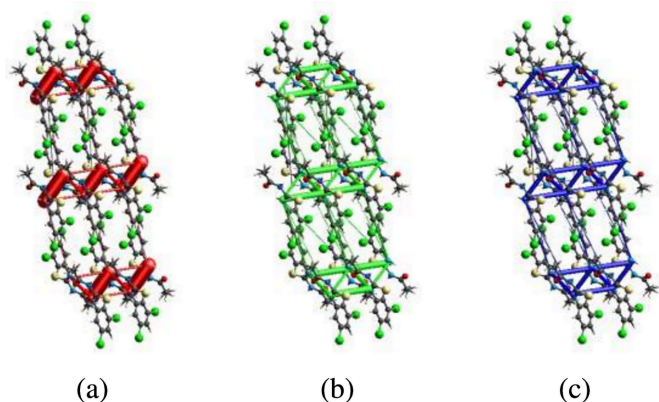


Figure 7

The energy frameworks for a cluster of molecules viewed down the *b* axis showing the (a) electrostatic energy, (b) dispersion energy and (c) total energy diagrams. The cylindrical radius is proportional to the relative strength of the corresponding energies and they were adjusted to the same scale factor of 80 with cut-off value of 5 kJ mol^{−1} within 2 × 2 × 2 unit cells.

(Turner *et al.*, 2015) with scale factors of 1.057, 0.740, 0.871 and 0.618, respectively (Mackenzie *et al.*, 2017).

Hydrogen bonding interaction energies (in kJ mol^{−1}) were calculated to be −7.9 (*E*_{ele}), −0.9 (*E*_{pol}), −16.2 (*E*_{dis}), 21.2 (*E*_{rep}) and −10.0 (*E*_{tot}) for the fragment N2–H2N...S1; −1.8 (*E*_{ele}), −0.2 (*E*_{pol}), −3.8 (*E*_{dis}), 2.5 (*E*_{rep}) and −3.9 (*E*_{tot}) for the fragment C9–H9F...S1; −2.6 (*E*_{ele}), −1.0 (*E*_{pol}), −6.1 (*E*_{dis}), 1.6 (*E*_{rep}) and −7.7 (*E*_{tot}) for the fragment C9–H9E...C11.

Energy frameworks combine the calculation of intermolecular interaction energies with a graphical representation of their magnitudes, in which they were constructed for *E*_{ele} (red cylinders), *E*_{dis} (green cylinders) and *E*_{tot} (blue cylinders) (Fig. 7*a–c*). The evaluation of the electrostatic, dispersion and total energy frameworks indicates that the stabilization in the crystal structure is dominated *via* the dispersion energy contributions.

4. Database survey

A search of the Cambridge Structural Database (CSD, Version 6.00, updated May 2025; Groom *et al.*, 2016) identified eleven structurally related structures of *N*-acetyl-*N'*-arylthiourea derivatives. These include: 3-acetyl-1-phenylthiourea, C₉H₁₀N₂OS (CSD refcode GARBIM; Shahwar *et al.*, 2012*a*), 3-acetyl-1-(2-methylphenyl)thiourea, C₁₀H₁₂N₂OS (YAXNUI; Shahwar *et al.*, 2012*b*), 3-acetyl-1-(3-chlorophenyl)thiourea, C₉H₉ClN₂OS (YAXTIC; Shahwar *et al.*, 2012*c*), 3-acetyl-1-(3-methylphenyl)thiourea, C₁₀H₁₂N₂OS (LEGQEV; Gowda *et al.*, 2012*a*), 3-acetyl-1-(2,5-dimethylphenyl)thiourea, C₁₁H₁₄N₂OS (LEDYAW; Gowda *et al.*, 2012*b*), 3-acetyl-1-(2,3-dichlorophenyl)thiourea, C₉H₈Cl₂N₂OS (LEFBEF; Gowda *et al.*, 2012*c*), 3-acetyl-1-(4-methylphenyl)thiourea, C₁₀H₁₂N₂OS (XAZYOO; Gowda *et al.*, 2012*d*), 3-acetyl-1-(2,4-dimethylphenyl)thiourea, C₁₁H₁₄N₂OS (LEGCIL; Gowda *et al.*, 2012*e*), 3-acetyl-1-(2,3-dimethylphenyl)thiourea, C₁₁H₁₄N₂OS (XEBKUM; Kumar *et al.*, 2012*a*), 3-acetyl-1-(2,6-dimethyl-

Table 2

Experimental details.

Crystal data	
Chemical formula	C ₉ H ₈ Cl ₂ N ₂ OS
<i>M</i> _r	263.13
Crystal system, space group	Triclinic, <i>P</i> $\bar{1}$
Temperature (K)	293
<i>a</i> , <i>b</i> , <i>c</i> (Å)	4.5764 (9), 10.873 (2), 12.093 (2)
α , β , γ (°)	102.87 (1), 92.65 (1), 99.04 (1)
<i>V</i> (Å ³)	577.27 (18)
<i>Z</i>	2
Radiation type	Mo <i>K</i> α
μ (mm ^{−1})	0.72
Crystal size (mm)	0.48 × 0.14 × 0.10
Data collection	
Diffractometer	Oxford Diffraction Xcalibur with Sapphire CCD
Absorption correction	Multi-scan (<i>CrysAlis RED</i> ; Oxford Diffraction, 2009)
<i>T</i> _{min} , <i>T</i> _{max}	0.725, 0.932
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	3301, 2007, 1747
<i>R</i> _{int}	0.013
(sin θ/λ) _{max} (Å ^{−1})	0.599
Refinement	
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.065, 0.139, 1.33
No. of reflections	2007
No. of parameters	144
No. of restraints	2
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ (e Å ^{−3})	0.46, −0.40

Computer programs: *CrysAlis CCD* (Oxford Diffraction, 2009), *CrysAlis RED* (Oxford Diffraction, 2009), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*), *ORTEP-3* for Windows (Farrugia, 2012), *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

phenyl)thiourea, C₁₁H₁₄N₂OS (LEDSE; Kumar *et al.*, 2012*b*), 3-acetyl-1-(2,6-dichlorophenyl)thiourea, C₉H₈Cl₂N₂OS (LEDSE; Kumar *et al.*, 2012*c*). Overall, these *N*-acetyl-*N'*-arylthiourea derivatives all exhibit hydrogen-bonding networks dominated by N–H...S and N–H...O interactions, which frequently generate graph-set motifs such as *R*₂²(8) and *R*₂²(12), and assemble the molecules into one-, two- or three-dimensional supramolecular networks. In several structures, three-center N–H...O(S) hydrogen bonds and weaker C–H...S contacts further reinforce the crystal packing, highlighting the key role of hydrogen-bonding in consolidating this family of compounds.

5. Synthesis and crystallization

Compound (**1**) was prepared by adding acetyl chloride (0.10 mol) dissolved in acetone (30 ml) dropwise to a suspension of potassium thiocyanate (0.10 mol) in acetone (30 ml). The resulting mixture was stirred for 2 h and subsequently refluxed for 30 min. After cooling to room temperature, a solution of 3,5-dichloroaniline (0.10 mol) in acetone (10 ml) was added, and the reaction mixture was refluxed for 3 h. The progress of the reaction mixture was monitored by thin-layer chromatography (TLC). Upon completion, the reaction mixture was poured into acidified cold water, resulting in the precipitation of the product. The solid material

was collected by suction filtration, thoroughly washed with water, and dried. Colourless crystals suitable for X-ray analysis were obtained by slow evaporation of an acetonitrile solution. Colourless solid, yield 80%, mp. 437 K. IR (KBr) cm^{-1} : 3183 (N–H str.), 1160 (C=S str.) and 1694 (C=O str.).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The hydrogen atoms of the methyl group were modelled over two sets of sites, with an occupancy ratio of 0.92 (6):0.08 (6). The NH hydrogen atoms were located from a difference Fourier map and refined with a distance restraint of 0.85 (2) Å. The C-bonded hydrogen atom positions were calculated geometrically at distances of 0.93 Å (for aromatic CH) and 0.96 Å (for methyl CH) and refined using a riding model by applying the constraint $U_{\text{iso}}(\text{H}) = k \times U_{\text{eq}}(\text{C}, \text{N})$, where $k = 1.5$ for methyl H and $k = 1.2$ for the other H atoms.

Acknowledgements

The authors thank the Yenepoya Deemed to be University for the facilities and financial support. TH is also grateful to Hacettepe University Scientific Research Project Unit (grant No. 013 D04 602 004). The authors' contributions are as follows. Conceptualization, SK and TH; synthesis, SK; X-ray analysis, SK and TH; Hirshfeld surface analysis, TH; writing (review and editing of the manuscript) SK and TH; supervision, TH and SK.

References

- Al-Abbassi, A. A., Kayed, S. F. & Kassim, M. B. (2023). *Inorg. Chem. Commun.* **156**, 111155.
- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2* **12**, S1–S19.
- Aly, A. A., Malah, T. E., Ishak, E. A., Brown, A. B. & Elayat, W. M. (2016). *J. Heterocycl. Chem.* **53**, 963–969.
- Asghar, F., Rana, S., Fatima, S., Badshah, A., Lal, B. & Butler, I. S. (2018). *New J. Chem.* **42**, 7154–7165.
- Bielenica, A., Kędzierska, E., Koliński, M., Kmiecik, S., Koliński, A., Fiorino, F., Severino, B., Magli, E., Corvino, A., Rossi, I., Massarelli, P., Kozioł, A. E., Sawczenko, A. & Struga, M. (2016). *Eur. J. Med. Chem.* **116**, 173–186.
- Blažek Bregović, V., Basarić, N. & Mlinarić-Majerski, K. (2015). *Coord. Chem. Rev.* **295**, 80–124.
- Canatar, C., Türkben, H., Efeoglu, C., Sari, H., Karasu, E., Nural, Y. & Ayaz, F. (2023). *ChemistrySelect* **8**, e202301258.
- Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* **B46**, 256–262.
- Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
- Gowda, B. T., Foro, S. & Kumar, S. (2012a). *Acta Cryst.* **E68**, o2574.
- Gowda, B. T., Foro, S. & Kumar, S. (2012b). *Acta Cryst.* **E68**, o2338.
- Gowda, B. T., Foro, S. & Kumar, S. (2012c). *Acta Cryst.* **E68**, o2353.
- Gowda, B. T., Foro, S. & Kumar, S. (2012d). *Acta Cryst.* **E68**, o2128.
- Gowda, B. T., Foro, S. & Kumar, S. (2012e). *Acta Cryst.* **E68**, o2498.
- Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* **B72**, 171–179.
- Guang, J., Larson, A. J. & Zhao, J. C. G. (2015). *Adv. Synth. Catal.* **357**, 523–529.
- Khan, K. M., Naz, F., Taha, M., Khan, A., Perveen, S., Choudhary, M. & Voelter, W. (2014). *Eur. J. Med. Chem.* **74**, 314–323.
- Kumar, S., Foro, S. & Gowda, B. T. (2012a). *Acta Cryst.* **E68**, o2306.
- Kumar, S., Foro, S. & Gowda, B. T. (2012b). *Acta Cryst.* **E68**, o2307.
- Kumar, S., Foro, S. & Gowda, B. T. (2012c). *Acta Cryst.* **E68**, o2191.
- Kumar, S. & Hökelek, T. (2026a). *Acta Cryst.* **E82**, 816–820.
- Kumar, S. & Hökelek, T. (2026b). *Acta Cryst.* **E82**, 1066–1070.
- Mackenzie, C. F., Spackman, P. R., Jayatilaka, D. & Spackman, M. A. (2017). *IUCrJ* **4**, 575–587.
- Mishra, A. & Batra, S. (2013). *Curr. Top. Med. Chem.* **13**, 2011–2025.
- Nammalwar, B., Berlin, K. D. & Bunce, R. A. (2013). *JSM Chem.* **1**, 1005.
- Oxford Diffraction (2009). *CrysAlis CCD and CrysAlis RED* Oxford Diffraction Ltd, Abingdon, England.
- Saeed, A., Flörke, U. & Erben, M. F. (2014). *J. Sulfur Chem.* **35**, 318–355.
- Saeed, A., Qamar, R., Fattah, T. A., Flörke, U. & Erben, M. F. (2017). *Res. Chem. Intermed.* **43**, 3053–3093.
- Shahwar, D., Tahir, M. N., Chohan, M. M., Ahmad, N. & Raza, M. A. (2012a). *Acta Cryst.* **E68**, o1160.
- Shahwar, D., Tahir, M. N., Chohan, M. M., Ahmad, N. & Raza, M. A. (2012c). *Acta Cryst.* **E68**, o1189.
- Shahwar, D., Tahir, M. N., Chohan, M. M., Ahmad, N. & Samiullah (2012b). *Acta Cryst.* **E68**, o508.
- Sheldrick, G. M. (2015a). *Acta Cryst.* **A71**, 3–8.
- Sheldrick, G. M. (2015b). *Acta Cryst.* **C71**, 3–8.
- Spackman, P. R., Turner, M. J., McKinnon, J. J., Wolff, S. K., Grimwood, D. J., Jayatilaka, D. & Spackman, M. A. (2021). *J. Appl. Cryst.* **54**, 1006–1011.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
- Turner, M. J., Thomas, S. P., Shi, M. W., Jayatilaka, D. & Spackman, M. A. (2015). *Chem. Commun.* **51**, 3735–3738.
- Yusof, M. S. M., Mutalib, S. F. A., Arshad, S. & Razak, I. A. (2012). *Acta Cryst.* **E68**, o982.

supporting information

Acta Cryst. (2026). E82 [https://doi.org/10.1107/S2056989026010121]

Synthesis, structure and computational study of 1-acetyl-3-(3,5-dichlorophenyl)thiourea

Sharatha Kumar and Tuncer Hökelek

Computing details

1-Acetyl-3-(3,5-dichlorophenyl)thiourea

Crystal data

$\text{C}_9\text{H}_8\text{Cl}_2\text{N}_2\text{OS}$

$M_r = 263.13$

Triclinic, $P\bar{1}$

$a = 4.5764$ (9) Å

$b = 10.873$ (2) Å

$c = 12.093$ (2) Å

$\alpha = 102.87$ (1)°

$\beta = 92.65$ (1)°

$\gamma = 99.04$ (1)°

$V = 577.27$ (18) Å³

$Z = 2$

$F(000) = 268$

$D_x = 1.514$ Mg m⁻³

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

Cell parameters from 1807 reflections

$\theta = 2.9\text{--}27.6^\circ$

$\mu = 0.72$ mm⁻¹

$T = 293$ K

Needle, colourless

$0.48 \times 0.14 \times 0.10$ mm

Data collection

Oxford Diffraction Xcalibur with Sapphire

CCD

diffractometer

Rotation method data acquisition using ω scans.

Absorption correction: multi-scan

(*CrysAlisRED*; Oxford Diffraction, 2009)

$T_{\min} = 0.725$, $T_{\max} = 0.932$

3301 measured reflections

2007 independent reflections

1747 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.013$

$\theta_{\max} = 25.2^\circ$, $\theta_{\min} = 2.9^\circ$

$h = -5 \rightarrow 5$

$k = -12 \rightarrow 13$

$l = -14 \rightarrow 14$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.065$

$wR(F^2) = 0.139$

$S = 1.33$

2007 reflections

144 parameters

2 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0189P)^2 + 1.3739P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.46$ e Å⁻³

$\Delta\rho_{\min} = -0.40$ e Å⁻³

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
C1	−0.1146 (11)	0.7042 (4)	0.2407 (4)	0.0450 (11)	
C2	0.0424 (11)	0.6056 (4)	0.2412 (4)	0.0450 (11)	
H2	0.074750	0.551893	0.173074	0.054*	
C3	0.1511 (10)	0.5877 (5)	0.3442 (4)	0.0463 (11)	
C4	0.1073 (12)	0.6672 (5)	0.4456 (4)	0.0531 (13)	
H4	0.183289	0.655586	0.514480	0.064*	
C5	−0.0512 (13)	0.7642 (5)	0.4430 (4)	0.0544 (14)	
C6	−0.1656 (12)	0.7838 (5)	0.3414 (4)	0.0518 (13)	
H6	−0.274366	0.849115	0.341043	0.062*	
C7	−0.1459 (10)	0.8188 (4)	0.0902 (3)	0.0380 (10)	
C8	−0.5341 (11)	0.7346 (5)	−0.0725 (4)	0.0477 (12)	
C9	−0.6596 (12)	0.7745 (5)	−0.1735 (4)	0.0566 (13)	
H9A	−0.504337	0.791366	−0.221559	0.085*	0.92 (6)
H9B	−0.743052	0.850490	−0.147804	0.085*	0.92 (6)
H9C	−0.811532	0.706967	−0.215545	0.085*	0.92 (6)
H9D	−0.868277	0.774516	−0.168380	0.085*	0.08 (6)
H9E	−0.629562	0.715392	−0.242135	0.085*	0.08 (6)
H9F	−0.561082	0.858915	−0.174394	0.085*	0.08 (6)
N1	−0.2353 (10)	0.7195 (4)	0.1336 (3)	0.0484 (10)	
N2	−0.2930 (9)	0.8207 (4)	−0.0129 (3)	0.0424 (9)	
O1	−0.6361 (9)	0.6373 (4)	−0.0442 (3)	0.0731 (12)	
Cl1	0.3434 (3)	0.46234 (15)	0.34501 (13)	0.0684 (4)	
Cl2	−0.1133 (5)	0.86361 (15)	0.57162 (12)	0.0870 (6)	
S1	0.1331 (3)	0.93861 (12)	0.14923 (10)	0.0476 (3)	
H1N	−0.376 (8)	0.662 (4)	0.095 (4)	0.057*	
H2N	−0.231 (11)	0.890 (3)	−0.033 (4)	0.057*	

Atomic displacement parameters ($\text{\AA}^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.054 (3)	0.039 (3)	0.036 (2)	−0.011 (2)	0.002 (2)	0.010 (2)
C2	0.052 (3)	0.042 (3)	0.033 (2)	−0.009 (2)	0.005 (2)	0.005 (2)
C3	0.047 (3)	0.048 (3)	0.040 (3)	−0.009 (2)	−0.002 (2)	0.013 (2)
C4	0.065 (3)	0.050 (3)	0.039 (3)	−0.012 (3)	−0.006 (2)	0.018 (2)
C5	0.082 (4)	0.045 (3)	0.029 (2)	−0.011 (3)	0.010 (2)	0.008 (2)
C6	0.069 (3)	0.042 (3)	0.043 (3)	0.001 (2)	0.007 (2)	0.012 (2)
C7	0.045 (3)	0.041 (2)	0.027 (2)	0.007 (2)	0.0044 (18)	0.0066 (18)
C8	0.054 (3)	0.049 (3)	0.035 (2)	−0.002 (2)	0.001 (2)	0.006 (2)
C9	0.057 (3)	0.062 (3)	0.046 (3)	0.001 (3)	−0.009 (2)	0.013 (3)

N1	0.062 (3)	0.045 (2)	0.034 (2)	−0.0096 (19)	−0.0047 (18)	0.0165 (18)
N2	0.047 (2)	0.041 (2)	0.038 (2)	0.0003 (18)	0.0022 (17)	0.0138 (17)
O1	0.089 (3)	0.065 (3)	0.053 (2)	−0.029 (2)	−0.017 (2)	0.0222 (19)
Cl1	0.0651 (9)	0.0730 (10)	0.0703 (9)	0.0123 (7)	−0.0036 (7)	0.0250 (8)
Cl2	0.1534 (17)	0.0627 (9)	0.0380 (7)	0.0055 (10)	0.0212 (9)	0.0037 (6)
S1	0.0534 (7)	0.0452 (7)	0.0416 (7)	−0.0070 (5)	−0.0059 (5)	0.0173 (5)

Geometric parameters (Å, °)

C1—C6	1.381 (7)	C7—N2	1.396 (5)
C1—C2	1.382 (7)	C7—S1	1.678 (4)
C1—N1	1.438 (6)	C8—O1	1.216 (6)
C2—C3	1.386 (6)	C8—N2	1.384 (6)
C2—H2	0.9300	C8—C9	1.502 (7)
C3—C4	1.377 (7)	C9—H9A	0.9600
C3—Cl1	1.736 (5)	C9—H9B	0.9600
C4—C5	1.376 (7)	C9—H9C	0.9600
C4—H4	0.9300	C9—H9D	0.9600
C5—C6	1.386 (7)	C9—H9E	0.9600
C5—Cl2	1.749 (5)	C9—H9F	0.9600
C6—H6	0.9300	N1—H1N	0.859 (19)
C7—N1	1.320 (6)	N2—H2N	0.858 (19)
C6—C1—C2	120.8 (5)	H9A—C9—H9B	109.5
C6—C1—N1	120.3 (5)	C8—C9—H9C	109.5
C2—C1—N1	118.9 (4)	H9A—C9—H9C	109.5
C1—C2—C3	119.2 (4)	H9B—C9—H9C	109.5
C1—C2—H2	120.4	C8—C9—H9D	109.5
C3—C2—H2	120.4	H9A—C9—H9D	141.1
C4—C3—C2	121.0 (5)	H9B—C9—H9D	56.3
C4—C3—Cl1	119.8 (4)	H9C—C9—H9D	56.3
C2—C3—Cl1	119.3 (4)	C8—C9—H9E	109.5
C5—C4—C3	118.7 (5)	H9A—C9—H9E	56.3
C5—C4—H4	120.7	H9B—C9—H9E	141.1
C3—C4—H4	120.7	H9C—C9—H9E	56.3
C4—C5—C6	121.7 (5)	H9D—C9—H9E	109.5
C4—C5—Cl2	118.8 (4)	C8—C9—H9F	109.5
C6—C5—Cl2	119.4 (4)	H9A—C9—H9F	56.3
C1—C6—C5	118.6 (5)	H9B—C9—H9F	56.3
C1—C6—H6	120.7	H9C—C9—H9F	141.1
C5—C6—H6	120.7	H9D—C9—H9F	109.5
N1—C7—N2	116.6 (4)	H9E—C9—H9F	109.5
N1—C7—S1	124.4 (3)	C7—N1—C1	123.9 (4)
N2—C7—S1	119.0 (3)	C7—N1—H1N	116 (4)
O1—C8—N2	122.8 (4)	C1—N1—H1N	120 (4)
O1—C8—C9	123.6 (4)	C8—N2—C7	127.8 (4)
N2—C8—C9	113.6 (4)	C8—N2—H2N	121 (4)
C8—C9—H9A	109.5	C7—N2—H2N	111 (4)

C8—C9—H9B	109.5		
C6—C1—C2—C3	−0.5 (7)	C4—C5—C6—C1	−0.8 (8)
N1—C1—C2—C3	−177.9 (4)	C12—C5—C6—C1	−179.9 (4)
C1—C2—C3—C4	−0.6 (7)	N2—C7—N1—C1	−178.1 (4)
C1—C2—C3—C11	179.0 (3)	S1—C7—N1—C1	3.3 (7)
C2—C3—C4—C5	1.0 (7)	C6—C1—N1—C7	68.6 (7)
C11—C3—C4—C5	−178.6 (4)	C2—C1—N1—C7	−113.9 (5)
C3—C4—C5—C6	−0.3 (8)	O1—C8—N2—C7	−5.9 (8)
C3—C4—C5—C12	178.8 (4)	C9—C8—N2—C7	173.0 (4)
C2—C1—C6—C5	1.2 (7)	N1—C7—N2—C8	4.2 (7)
N1—C1—C6—C5	178.6 (4)	S1—C7—N2—C8	−177.1 (4)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
N1—H1N $\cdots$ O1	0.86 (2)	1.95 (4)	2.655 (5)	138 (5)
N2—H2N $\cdots$ S1 ⁱ	0.86 (2)	2.57 (2)	3.395 (4)	163 (5)
C9—H9F $\cdots$ S1 ⁱ	0.96	2.66	3.576 (5)	160
C9—H9E $\cdots$ C11 ⁱⁱ	0.96	2.62	3.478 (6)	149

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