

1,3-Bis(2-chlorobenzoyl)-3,4,5,6-tetrahydro-  
pyrimidine-2(1H)-thioneSema Öztürk Yıldırım,<sup>a</sup> Mehmet Akkurt,<sup>a\*</sup> Murat Genç,<sup>b</sup> Memet Şekerci<sup>b</sup> and H.-K. Fun<sup>c</sup><sup>a</sup>Department of Physics, Faculty of Arts and Sciences, Erciyes University, 38039 Kayseri, Turkey, <sup>b</sup>Department of Chemistry, Faculty of Arts and Sciences, Firat University, 23119 Elazığ, Turkey, and <sup>c</sup>X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM Penang, Malaysia

Correspondence e-mail: akkurt@erciyes.edu.tr

## Key indicators

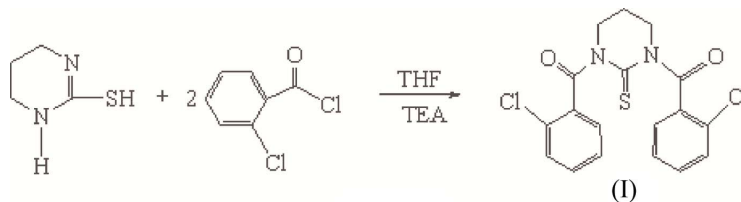
Single-crystal X-ray study  
*T* = 293 K  
Mean  $\sigma(\text{C}-\text{C}) = 0.005 \text{ \AA}$   
Disorder in main residue  
*R* factor = 0.053  
*wR* factor = 0.186  
Data-to-parameter ratio = 24.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound,  $\text{C}_{18}\text{H}_{14}\text{Cl}_2\text{N}_2\text{O}_2\text{S}$ , was synthesized by the reaction of 1,4,5,6-tetrahydropyrimidine-2-thiol and 2-chlorobenzoyl chloride. The dihedral angle between the two chlorobenzene rings is  $76.2(1)^\circ$ . The ring carbon (and attached H atoms) opposite the  $\text{C}=\text{S}$  group are disordered over two sites. In the crystal structure, there is one intermolecular  $\text{C}-\text{H}\cdots\text{O}$  and one intramolecular  $\text{C}-\text{H}\cdots\text{N}$  hydrogen bond.

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## Comment

Pyrimidine and 1,3-diazepine derivatives are used in industry and in the pharmaceutical field. Saturated pyrimidine derivatives are useful as transport molecules for tumor inhibition, as well as antidepressants and sedatives. Certain fully saturated derivatives of pyrimidine have other industrial applications, *e.g.* as antioxidants and catalysts (Marino *et al.*, 2001; Zaleska *et al.*, 2002). Heterocyclic thioamides, usually occurring in their thioketo form, are referred to as 'thiones'; these compounds are of particular interest in coordination chemistry because they display both hard and soft donor sites (Kazak *et al.*, 2005). Only a few methods for the synthesis of imidazoline-2-thione and its homologous heterocyclic derivatives have been reported in the literature (Billman & Khan, 1968; Szinai & Crank, 1970; Matsuhisa *et al.*, 1998). In this study, the title compound, (I), was obtained by the reaction of 1,4,5,6-tetrahydropyrimidine-2-thiol and 2-chlorobenzoyl chloride. The structure of (I) was suggested by IR and  $^1\text{H}$  NMR spectroscopic analyses. In order to confirm the assigned structure, we undertook this X-ray crystallographic analysis.



The molecular structure of (I) is shown in Fig. 1, with the atom-numbering scheme. The fragments *A* (atoms C1–C6/Cl1) and *B* (atoms C13–C18/Cl2) are each almost planar, with a maximum deviation of  $0.028(2) \text{ \AA}$  for atom C6 in *A*, and  $0.020(1) \text{ \AA}$  for Cl2 in *B*. These planes make a dihedral angle of  $76.2(1)^\circ$  with each other. The values of the geometric parameters in (I) are in good agreement with those found for other similar structures (Allen *et al.*, 1987; Kazak *et al.*, 2005).

The structure of (I) is stabilized by intermolecular  $\text{C}-\text{H}\cdots\text{O}$  and intramolecular  $\text{C}-\text{H}\cdots\text{N}$  hydrogen-bonding interactions (Table 1 and Fig. 2).

Experimental

1,4,5,6-Tetrahydropyrimidine-2-thiol (9.8 mmol) was dissolved in 100 ml tetrahydrofuran (THF) and 2.71 ml triethylamine (19.6 mmol) was added. The reaction mixture was stirred on an ice bath for 0.5 h. To the mixture, 2-chlorobenzoyl chloride (19.6 mmol, 2.5 ml) was added dropwise over a period of 10 m and the mixture was refluxed for 4 h. The solution was then evaporated under vacuum to half its volume and poured into ice–water, resulting in a precipitate. The residue was filtered off and recrystallized from acetone (yield 81%, m.p. 493 K).

Crystal data

C<sub>18</sub>H<sub>14</sub>Cl<sub>2</sub>N<sub>2</sub>O<sub>2</sub>S  
 M<sub>r</sub> = 393.28  
 Monoclinic, P2<sub>1</sub>/c  
 a = 11.361 (5) Å  
 b = 12.917 (5) Å  
 c = 12.186 (5) Å  
 β = 95.399 (5)°  
 V = 1780.4 (13) Å<sup>3</sup>

Z = 4  
 D<sub>x</sub> = 1.467 Mg m<sup>-3</sup>  
 Mo Kα radiation  
 μ = 0.50 mm<sup>-1</sup>  
 T = 293 K  
 Block, translucent dark green  
 0.66 × 0.64 × 0.64 mm

Data collection

Siemens SMART CCD area-detector diffractometer  
 φ and ω scans  
 Absorption correction: none  
 20918 measured reflections

5828 independent reflections  
 3082 reflections with I > 2σ(I)  
 R<sub>int</sub> = 0.034  
 θ<sub>max</sub> = 31.7°

Refinement

Refinement on F<sup>2</sup>  
 R[F<sup>2</sup> > 2σ(F<sup>2</sup>)] = 0.053  
 wR(F<sup>2</sup>) = 0.186  
 S = 1.08  
 5828 reflections  
 236 parameters  
 H-atom parameters constrained

w = 1/[σ<sup>2</sup>(F<sub>o</sub><sup>2</sup>) + (0.0706P)<sup>2</sup> + 0.7185P]  
 where P = (F<sub>o</sub><sup>2</sup> + 2F<sub>c</sub><sup>2</sup>)/3  
 (Δ/σ)<sub>max</sub> < 0.001  
 Δρ<sub>max</sub> = 0.43 e Å<sup>-3</sup>  
 Δρ<sub>min</sub> = -0.24 e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

D—H...A	D—H	H...A	D...A	D—H...A
C9—H9A...O2 <sup>i</sup>	0.97	2.47	3.317 (4)	146
C18—H18...N2	0.93	2.59	2.914 (4)	101

Symmetry code: (i) x, -y + ½, z - ½.

All H atoms were placed in calculated positions, with C—H = 0.93–0.97 Å, and refined as riding. U<sub>iso</sub>(H) values were set equal to 1.2U<sub>eq</sub>(C). Atom C10, together with its attached H atoms, is disordered over two sites, A and B; the site-occupancy factors refined to 0.52 (2) and 0.48 (2), respectively.

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SIR97 (Altomare *et al.*, 1999); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: WinGX (Farrugia, 1999).

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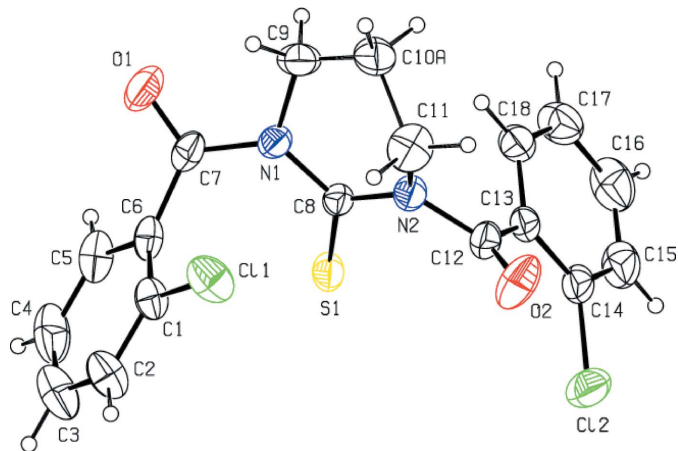


Figure 1

The molecular structure of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small circles of arbitrary radii. The minor disorder component is not shown.

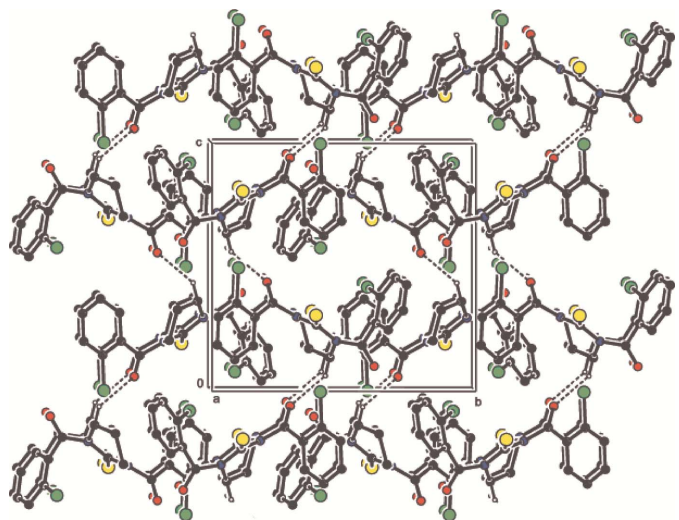


Figure 2

The crystal packing of (I), projected on to the bc plane. Dashed lines indicate hydrogen bonds. The minor disorder component and H atoms not involved in hydrogen bonding have been omitted.

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