

## 1-(2,4-Dichlorophenyl)-3-(2-thienyl)prop-2-en-1-one

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## Key indicators

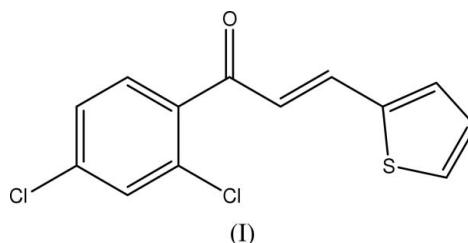
Single-crystal X-ray study  
 $T = 100\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.001\text{ \AA}$   
Disorder in main residue  
 $R$  factor = 0.026  
 $wR$  factor = 0.079  
Data-to-parameter ratio = 41.5For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The thiophene ring of the title compound,  $\text{C}_{13}\text{H}_8\text{Cl}_2\text{OS}$ , is disordered. The disorder corresponds to an approximate  $180^\circ$  rotation of the thiophene ring with respect to the C—C bond linking it to the rest of the molecule. The crystal packing is stabilized by short  $\text{Cl}\cdots\text{Cl}$  and  $\text{S}\cdots\text{O}$  contacts, together with intermolecular  $\text{C}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\text{S}$  interactions.

Received 13 September 2006  
Accepted 15 September 2006

## Comment

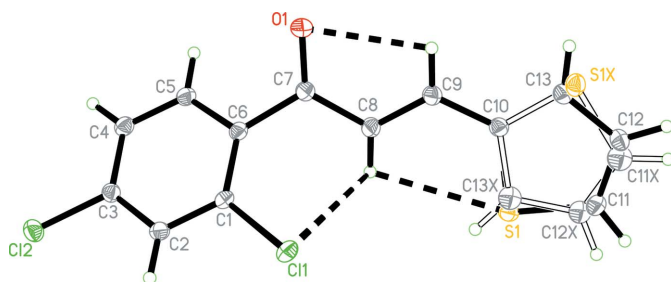
Many chalcone derivatives crystallize in non-centrosymmetric space groups and display significant nonlinear optical (NLO) properties (Uchida *et al.*, 1998). As part of our study on NLO chalcone derivatives (Patil, Teh *et al.*, 2006*a,b,c*), we report here the synthesis and crystal structure of the title compound, (I). Crystals of (I) do not exhibit second-order NLO properties as they crystallize in a centrosymmetric space group.



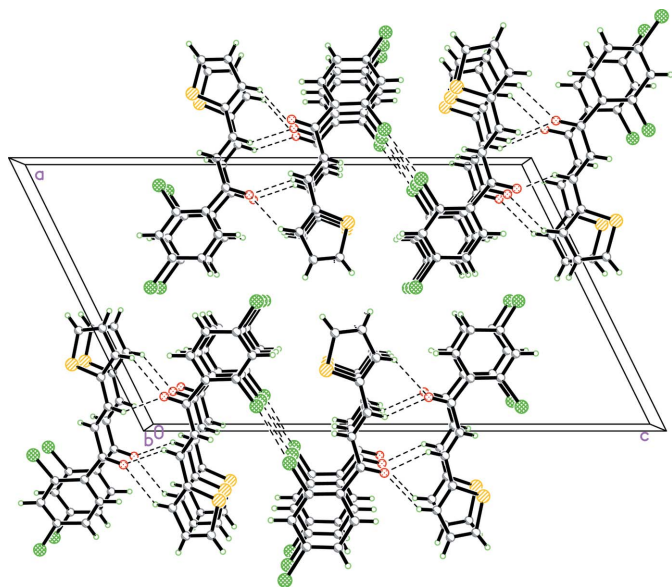
In (I) (Fig. 1), the thiophene ring is disordered over two sites (atoms of the minor occupancy component are labelled with the suffix *X*). The disorder corresponds to an approximate  $180^\circ$  rotation of the thiophene ring with respect to the C9—C10 bond. Bond lengths and angles in (I) are comparable to those reported for related structures (Ng *et al.*, 2006; Rosli *et al.*, 2006; Patil, Ng *et al.*, 2006; Teh *et al.*, 2006) and show normal values (Allen *et al.*, 1987).

The benzene ring and thiophene rings are individually planar. The molecule is slightly twisted about the C7—C6 bond, with dihedral angles of  $36.56(6)$  and  $36.9(2)^\circ$  between the benzene ring and rings C10/S1/C11—C13 and C10/S1X/C11X—C13X, respectively.

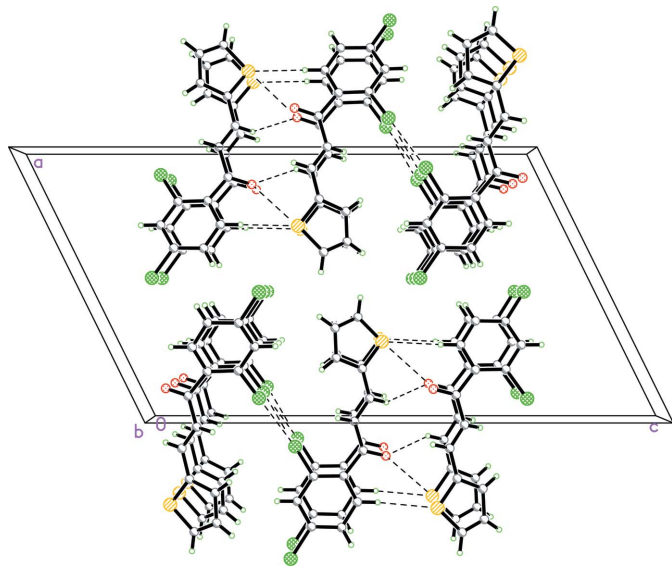
The intramolecular C8—H8A $\cdots$ C11 interaction generates an *S*(6) ring motif, while the C8—H8A $\cdots$ S1 and C9—H9A $\cdots$ O1 intramolecular interactions (Fig. 1) generate *S*(5) ring motifs (Bernstein *et al.*, 1995). In the crystal structure, the molecules propagate down the *b* axis and are stabilized by two short  $\text{Cl}\cdots\text{Cl}$  contacts [ $3.4705(3)\text{ \AA}$ ], by which each molecule is linked to others related by the symmetry operations  $(-x, -\frac{1}{2} + y, \frac{1}{2} - z)$  and  $(-x, \frac{1}{2} + y, \frac{1}{2} - z)$  (Figs. 2 and 3). The intermolecular C9—H9A $\cdots$ O1<sup>ii</sup> (Figs. 2 and 3) and C13—H13A $\cdots$ O1<sup>ii</sup> (Fig. 2) interactions (Table 1) form a pair of



**Figure 1**  
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atomic numbering. Dashed lines indicate intramolecular hydrogen bonds. Both disorder components are shown.



**Figure 2**  
Packing of the major component of the crystal structure of (I), viewed down the *b* axis. Dashed lines indicate short Cl...Cl contacts and intermolecular C—H...O interactions.



**Figure 3**  
Packing of the minor component of the crystal structure of (I), viewed down the *b* axis. Dashed lines indicate short Cl...Cl and S...O contacts and intermolecular C—H...O and C—H...S interactions.

bifurcated acceptor bonds, generating a ring of graph-set motif  $R_2^1(6)$  (Bernstein *et al.*, 1995). In addition, there is a short  $S1X \cdots O1^{iii}$  contact of 3.264 (4) Å [symmetry code: (iii)  $-x, -y + 1, -z$ ] between molecules arranged in the [011] direction (Fig. 3). The weak intermolecular  $C5-H5A \cdots S1X^i$  interaction (Table 1) further stabilizes the crystal packing (Fig. 3).

## Experimental

2-Thiophenecarbaldehyde (0.1 mol) and 2,4-dichloroacetophenone (0.1 mol) were stirred in 100 ml of ethanol at 298 K. An aqueous NaOH solution (10 g, 10%) was added and the mixture was stirred for 2 h. The precipitate that formed was filtered off, washed with water and dried. The resulting crude product was recrystallized twice from acetone. Crystals suitable for single-crystal X-ray diffraction experiments were grown by slow evaporation of an acetone solution of (I).

### Crystal data

$C_{13}H_8Cl_2OS$	$Z = 4$
$M_r = 283.15$	$D_x = 1.577 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 14.1918$ (2) Å	$\mu = 0.70 \text{ mm}^{-1}$
$b = 3.8515$ (1) Å	$T = 100.0$ (1) K
$c = 24.3418$ (3) Å	Block, yellow
$\beta = 116.283$ (1)°	$0.62 \times 0.59 \times 0.33 \text{ mm}$
$V = 1192.96$ (4) Å <sup>3</sup>	

### Data collection

Bruker SMART APEX2 CCD area-detector diffractometer	50586 measured reflections
$\omega$ scans	7306 independent reflections
Absorption correction: multi-scan (SADABS; Bruker, 2005)	6624 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.716$ , $T_{\max} = 0.805$	$R_{\text{int}} = 0.024$
	$\theta_{\max} = 40.0^\circ$

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0413P)^2 + 0.2466P]$
$R[F^2 > 2\sigma(F^2)] = 0.026$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.080$	$(\Delta/\sigma)_{\max} < 0.001$
$S = 1.10$	$\Delta\rho_{\max} = 0.61 \text{ e \AA}^{-3}$
7306 reflections	$\Delta\rho_{\min} = -0.28 \text{ e \AA}^{-3}$
176 parameters	
H-atom parameters constrained	

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$C8-H8A \cdots Cl1$	0.93	2.75	3.123 (1)	105
$C8-H8A \cdots S1$	0.93	2.83	3.185 (1)	104
$C9-H9A \cdots O1$	0.93	2.50	2.823 (1)	101
$C5-H5A \cdots S1X^i$	0.93	2.89	3.787 (4)	162
$C9-H9A \cdots O1^{ii}$	0.93	2.54	3.362 (1)	148
$Cl3-H13A \cdots O1^{ii}$	0.93	2.54	3.327 (3)	143

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

H atoms were placed in calculated positions and constrained to ride on their carrier atoms, with  $C-H = 0.93 \text{ \AA}$  and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ . The ratio of the refined site occupancies for the major and minor components of the disordered thiophene ring are 0.8399 (12):0.1601 (12). Similarity and rigid-bond restraints were applied to the disordered atoms.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

The authors thank the Malaysian Government and Universiti Sains Malaysia for the Scientific Advancement Grant Allocation (SAGA) grant No. 304/PFIZIK/653003/A118. PSP and SMD are grateful to DRDO, the Government of India, for financial assistance.

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