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1-(4-Chlorophenyl)-3-(2-thienyl)prop-2-en-1-one

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.001 Å
Disorder in main residue
R factor = 0.031
wR factor = 0.089
Data-to-parameter ratio = 33.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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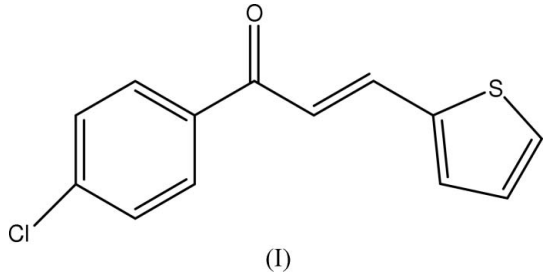
The enone fragment, the thiophene ring and the benzene ring of the title molecule, $C_{13}H_9ClOS$, are all essentially individually planar. The thiophene ring is disordered over two sites, corresponding to a rotation of approximately 180° about the single C—C bond to which it is attached. The crystal packing is stabilized by weak intermolecular C—H···π interactions involving thiophene rings and benzene rings, and molecules are stacked along the *b* axis.

Comment

Chalcones are not only of interest due to their pharmacological activities (De Vincenzo *et al.*, 1995; Kumar *et al.*, 2003) but also because of their extremely high and fast non-linearity (Fichou *et al.*, 1988; Kitaoka *et al.*, 1990; Uchida *et al.*,1998; Goto *et al.*, 1991; Patil *et al.*, 2006*a*,*b*; Zhang *et al.*, 1990; Zhao *et al.*, 2000). The single-crystal X-ray structural study of the title compound, (I), was undertaken in order to establish the molecular structure, and the results are presented here. The crystallization of (I) in a centrosymmetric space group precludes the presence of second-order non-linear optical properties.



(I)

In the title molecular structure (Fig. 1), the thiophene ring is disordered over two sites (minor and major components labelled with suffixes *A* and *B*, respectively), corresponding to a rotation of approximately 180° about the single C—C bond to which it is attached. The bond lengths and angles are normal (Allen *et al.*, 1987) and comparable with those in related structures (Ng *et al.*, 2006; Patil *et al.*, 2006*a*,*b*), with the exception of the bond lengths S1*A*—C10 [1.685 (1) Å], S1*A*—C11*A* [1.438 (5) Å], C10═C13*A* [1.649 (7) Å], C12—C13*A* [1.687 (2) Å], C13*B*—C12*B* [1.646 (7) Å] and C10—C13*B* [1.424 (4) Å], which is probably due to systematic errors relating to the disorder in the thiophene ring. The chlorophenyl and thiophene rings are individually planar, with the largest deviations of 0.010 (1), 0.025 (6) and 0.020 (6) Å for atoms C1, C11*A* and C13*B*, respectively. The molecule is slightly twisted about the C6—C7 bond, with a dihedral angle

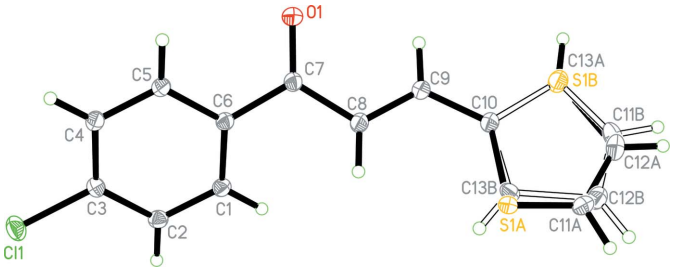


Figure 1
The title molecular structure, showing 50% probability displacement ellipsoids and small sheres for H atoms. Open bonds correspond to the major component of the disorder.

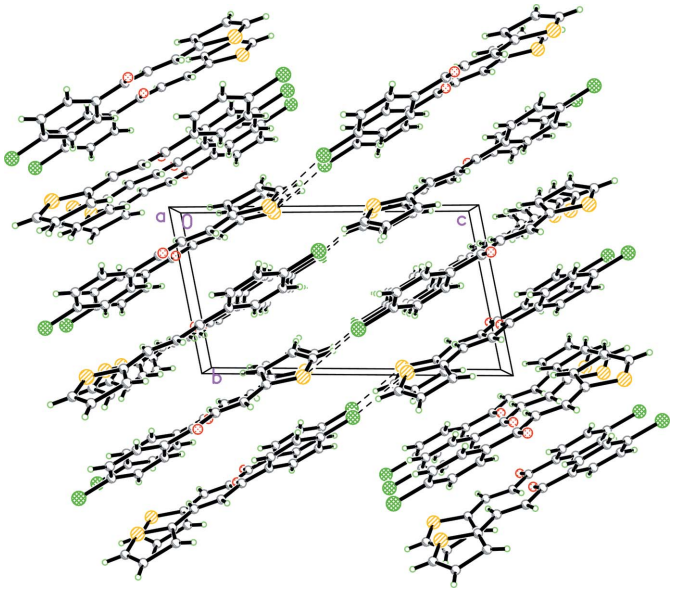


Figure 2
Part of the crystal structure of (I). Dashed lines indicate close S···Cl contacts. The minor component of the disorder has been omitted.

of 47.0 (3)° between the benzene ring and the ring C10/S1*A*/C11*A*–C13*A*, and 47.4 (2)° for C10/S1*B*/C11*B*–C13*B*.

The crystal structure of (I) is stabilized by weak intermolecular C—H···π interactions involving the thiophene and benzene rings, and details are listed in Table 1. The closest intermolecular H···S contact of 2.89 Å for H5*A*···S1*A* [C5···S1*A* = 3.718 (4) Å and C5—H5*A*···S1*A* = 149°] probably falls outside the range for a significant C—H···S hydrogen bond, but there is a relatively short contact of 3.4583 (3) Å for S1*B*—Cl1^{v} [symmetry code: (v) x, $-1+y$, $1+z$] between molecules arranged as one-dimensional chains along [0$\bar{1}$1] (Fig. 2).

Experimental

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

Crystal data

$C_{13}H_9ClOS$
M_r = 248.71
Triclinic, $P\bar{1}$
a = 5.7956 (1) Å
b = 7.3397 (1) Å
c = 13.3905 (2) Å
α = 78.634 (1)°
β = 81.928 (1)°
γ = 88.964 (1)°
V = 552.87 (1) Å^3
Z = 2
D_x = 1.494 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.51 mm^{-1}
T = 100.0 (1) K
Block, yellow
0.56 × 0.54 × 0.23 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.810, T_{max} = 0.892
28531 measured reflections
5770 independent reflections
5316 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 37.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.089
S = 1.06
5770 reflections
173 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0455P)^2 + 0.1565P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.96 e Å^{-3}
$\Delta\rho_{min}$ = −0.86 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

*Cg*1, *Cg*2 and *Cg*3 are the centroids of rings C10/S1*B*/C11–C13*B*, C10/S1*A*/C11–C13*A* and C1–C6, respectively.

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2*A*···*Cg*1^{i}	0.93	2.90	3.559 (3)	129
C2—H2*A*···*Cg*2^{i}	0.93	2.90	3.538 (3)	127
C5—H5*A*···*Cg*1^{ii}	0.93	2.84	3.467 (3)	126
C5—H5*A*···*Cg*2^{ii}	0.93	2.82	3.475 (3)	128
C9—H9*A*···*Cg*3^{iii}	0.93	2.98	3.528 (1)	119
C12*B*—H12*C*···*Cg*3^{iv}	0.93	2.87	3.518 (6)	128
C11*A*—H11*A*···*Cg*3^{iv}	0.93	2.89	3.501 (6)	124

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

H atoms were placed in calculated positions and constrained to ride on their carrier atoms, with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C). The refined occupancies for the major and minor components of the disordered thiophene ring are 0.5198 (12) and 0.4802 (12).

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; 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*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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