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

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The enone fragment, thiophene ring and benzene ring of the title molecule, $C_{13}H_9BrOS$, are each essentially 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 the thiophene and benzene rings, and molecules are stacked along the b axis.

Comment

Non-linear optical (NLO) materials capable of generating second-harmonic frequencies play an important role in the domain of opto-electronics and photonics. NLO crystals with high conversion efficiencies for second-harmonic generation (SHG) and which are transparent in the visible and ultraviolet ranges are required for numerous device applications. Within the last decade, much progress has been made in the development of these NLO organic materials having large non-linear optical coefficients. Chalcone derivatives are organic compounds with excellent NLO properties (Fichou *et al.*, 1988; Kitaoka *et al.*, 1990; Uchida *et al.*, 1998; Goto *et al.*, 1991; Patil *et al.*, 2006a,b; Zhang *et al.*, 1990; Zhao *et al.*, 2000). Furthermore, chalcones show interesting biological and pharmacological activities (De Vincenzo *et al.*,1995; Kumar *et al.*, 2003). The single-crystal X-ray structural study of the title compound, (I), was undertaken in order to establish the structure and conformation of the various groups. Crystallization of the title compound in a centrosymmetric space group precludes second-order non-linear optical properties.

(I)
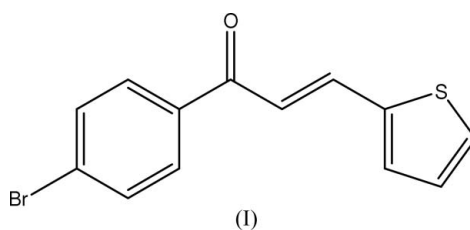
In the title molecular structure (Fig. 1), the thiophene ring is disordered over two sites (atoms of the minor occupancy component are labelled with the suffix *X*), corresponding to a rotation of approximately 180° about C9—C10. The bond lengths and angles observed are normal (Allen *et al.*, 1987) and agree with those in related structures (Ng *et al.*, 2006; Patil *et al.*, 2006a,b), with the exception of some parameters of the thiophene ring, which are probably a consequence of the disorder. The benzene and thiophene rings are individually planar, with maximum deviations of 0.009 (1) Å for atom C2 and 0.013 (12) Å for C13*X*. The molecule is twisted about the
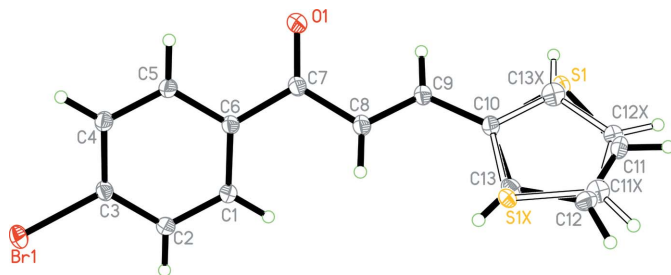
Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atomic numbering. Open bonds indicate the minor disorder component.
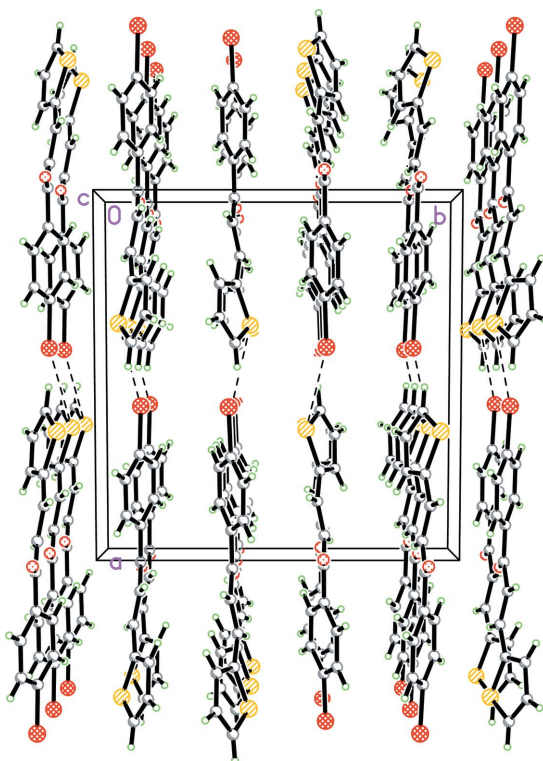

Figure 2
Part of the crystal structure of (I), viewed along the *c* axis. The dashed lines indicate short S···Br contacts. The minor disorder component is not shown.

C6—C7 bond with a dihedral angle of 46.49 (11)° between the benzene ring and C10/S1/C11–C13 [48.4 (3)° for C10/S1*X*/ C11*X*–C13*X*].

The crystal structure is stabilized by weak intermolecular C—H···π interactions involving the thiophene ring and benzene ring; details are given in Table 1. The closest intermolecular H···Br contact of 3.06 Å for H11*B*···Br1 (C11*X*— H11*B* = 0.93 Å, C11*X*···Br1 = 3.776 (8) Å and C11*X*— H11*B*···Br1 = 135°) probably falls outside the range for significant C—H···Br hydrogen bonds, but there is a relatively short Br1···S1^v contact of 3.4596 (6) Å [symmetry code: (v) 1 + *x*, *y*, *z*] between molecules arranged as one-dimensional chains along [100] (Fig. 2). In addition, molecules propagating along the *c*-axis direction are involved in fairly short S···S contacts [S1*X*···S1*X*($x, \frac{3}{2} - y, \frac{1}{2} + z$) = 3.653 (8) Å].

Experimental

2-Thiophenecarboxyaldehyde (0.1 mol) and 4-bromoacetophenone (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 formed was filtered off, washed with water and dried. The resulting crude product was recrystallized twice from acetone. The purity of the compound was checked by thin-layer chromatography. Crystals suitable for single-crystal X-ray diffraction experiments were grown by slow evaporation of an acetone solution.

Crystal data

$C_{13}H_9BrOS$	$Z = 4$
$M_r = 293.17$	$D_x = 1.732$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 14.0993$ (3) Å	$\mu = 3.81$ mm^{-1}
$b = 13.8355$ (3) Å	$T = 100.0$ (1) K
$c = 5.8022$ (1) Å	Block, yellow
$\beta = 96.634$ (1)°	$0.44 \times 0.42 \times 0.38$ mm
$V = 1124.26$ (4) Å^3	

Data collection

Bruker SMART APEX-2 CCD area-detector diffractometer	64573 measured reflections
ω scans	6876 independent reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	5478 reflections with $I > 2\sigma(I)$
$T_{min} = 0.204$, $T_{max} = 0.234$	$R_{int} = 0.044$
	$\theta_{max} = 40.0°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0367P)^2 + 0.474P]$
$R[F^2 > 2\sigma(F^2)] = 0.029$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.076$	$(\Delta/\sigma)_{max} = 0.003$
$S = 1.03$	$\Delta\rho_{max} = 1.36$ e Å^{-3}
6876 reflections	$\Delta\rho_{min} = -0.58$ e Å^{-3}
168 parameters	Extinction correction: *SHELXL97*
H-atom parameters constrained	Extinction coefficient: 0.0100 (7)

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

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2*A*···*Cg*1^i	0.93	2.90	3.546 (2)	128
C2—H2*A*···*Cg*2^i	0.93	2.91	3.558 (4)	128
C5—H5*A*···*Cg*3^{ii}	0.93	2.98	3.507 (1)	118
C9—H9*A*···*Cg*3^{iii}	0.93	2.94	3.504 (1)	120
C12—H12*A*···*Cg*1^{iv}	0.93	2.88	3.528 (2)	128
C12—H12*A*···*Cg*2^{iv}	0.93	2.86	3.508 (4)	128

Symmetry codes: (i) $-x, -y + 1, -z$; (ii) $x, -y + \frac{3}{2}, z + \frac{1}{2}$; (iii) $-x, -y + 1, -z + 1$; (iv) $x, -y + \frac{3}{2}, z - \frac{1}{2}$.

H atoms were placed in calculated positions, with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C). The ratio of the refined occupancies for the major and minor components of the disordered thiophene ring is 0.791 (2):0.209 (2). Similarity and rigid-bond restraints were applied to the disordered atoms. The largest peak in the final difference Fourier map is located 0.68 Å from atom Br1.

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

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