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3-(5-Bromo-2-thienyl)-1-(4-methoxyphenyl)-prop-2-en-1-one

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Key indicators

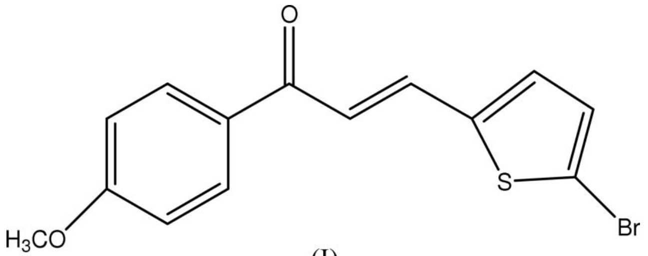
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.003 Å
R factor = 0.035
wR factor = 0.098
Data-to-parameter ratio = 39.4

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

The title molecule, $C_{18}H_{17}ClO_4$, deviates slightly from planarity with a dihedral angle of 27.18 (11)° between the rings. The crystal structure is stabilized by intermolecular C—H···O and C—H···Br interactions.

Comment

The second-harmonic generation (SHG) conversion efficiency of the title compound, (I) (Fig. 1), in powder form was reported earlier by Goto *et al.* (1991) (26 times that of urea, *Q*-switched Nd$^+$–YAG laser, pulse width of 10 ns, repetition frequency of 10 Hz and wavelength of 1064 nm). However, its single-crystal structure has not yet been reported. We report here the details of the molecular and supramolecular structure of (I), which crystallizes in a non-centrosymmetric space group, thus satisfying one of the basic and essential requirements for SHG activity in the material.



(I)

Bond lengths and angles in (I) are comparable with those reported for related structures (Ng *et al.*, 2006; Patil *et al.*, 2006) and display normal values (Allen *et al.*, 1987). The benzene ring and thiophene ring are each planar. The molecule is twisted about the C7—C8 bond, with a dihedral angle of 27.18 (11)° between the benzene and thiophene rings.

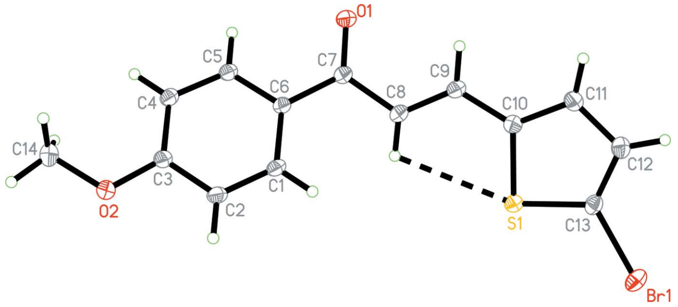


Figure 1
The molecule structure of (I), showing 50% probability displacement ellipsoids and the atomic numbering. An intramolecular hydrogen bonds is shown as a dashed line.

An intramolecular C8—H8*A*···S1 interaction generates an *S*(5) ring motif (Bernstein *et al.*, 1995). In the crystal structure, the molecules propagate down the *c* axis with screw-related molecules in adjacent columns which are connected by intermolecular C11—H11*A*···O2^{i} and C14—H14*A*···O1^{ii} interactions. Adjacent columns of molecules are further stabilized by weak intermolecular C1—H1*A*···Br1^{iii} interactions (Fig. 2 and Table 1).

Experimental

5-Bromothiophene-2-carbaldehyde (0.01 mol) and 4-methoxyacetophenone (0.01 mol) were stirred in 60 ml of methanol at room temperature. 5 g of 10% NaOH aqueous solution was added and the mixture was stirred for 2 h. The precipitate was filtered, washed with water, dried and the crude product recrystallized twice from acetone. Crystals suitable for X-ray analysis were grown by slow evaporation of an acetone solution at room temperature.

Crystal data

$C_{14}H_{11}BrO_2S$
$M_r = 323.20$
Orthorhombic, $Pna2_1$
$a = 10.1774$ (2) Å
$b = 31.8924$ (6) Å
$c = 3.9078$ (1) Å
$V = 1268.40$ (5) Å^3
$Z = 4$
$D_x = 1.692$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 3.40$ mm^{-1}
$T = 100.0$ (1) K
Block, yellow
0.63 × 0.47 × 0.39 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.222$, $T_{max} = 0.353$ (expected range = 0.168–0.266)
27460 measured reflections
6420 independent reflections
5576 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$
$\theta_{max} = 37.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.099$
$S = 1.11$
6420 reflections
163 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0488P)^2 + 0.3039P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.81$ e Å^{-3}
$\Delta\rho_{min} = -0.70$ e Å^{-3}
Absolute structure: Flack (1983), 2706 Friedel pairs
Flack parameter: 0.092 (7)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8*A*···S1	0.93	2.78	3.168 (2)	106
C11—H11*A*···O2^{i}	0.93	2.57	3.383 (2)	146
C14—H14*A*···O1^{ii}	0.96	2.55	3.460 (2)	158
C1—H1*A*···Br1^{iii}	0.93	2.97	3.358 (2)	107

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

H atoms were placed in calculated positions, with C—H distances in the range 0.93–0.96 Å. The U_{iso} values were were set equal to 1.5U_{eq} of the carrier atom for methyl H atoms and 1.2U_{eq} for the remaining H atoms.

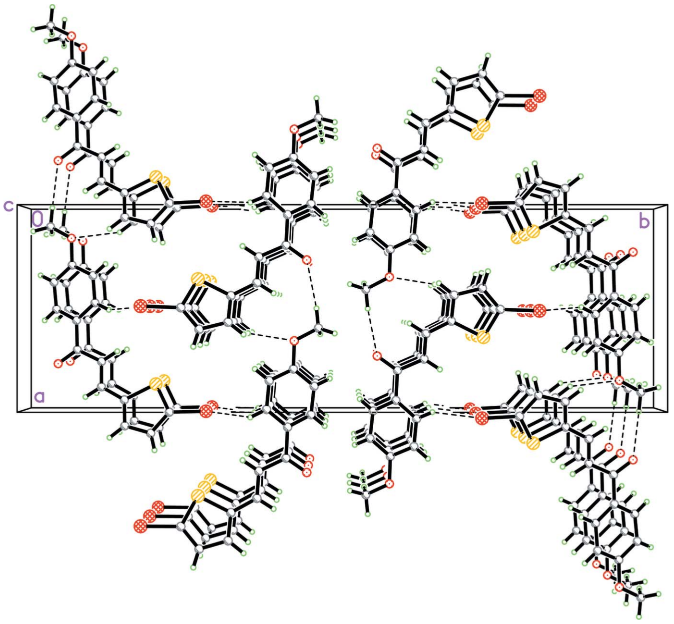


Figure 2
The packing of (I), viewed down the *c* axis. Dashed lines indicate hydrogen bonds.

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*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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