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

P. S. Patil,[a] Mohd Mustaqim Rosli,[b] Hoong-Kun Fun,[b]* Ibrahim Abdul Razak[b] and S. M. Dharmaprakash[a]

[a]Department of Studies in Physics, Mangalore University, Mangalagangotri, Mangalore 574 199, India, and [b]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia

Correspondence e-mail: hkfun@usm.my

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Key indicators

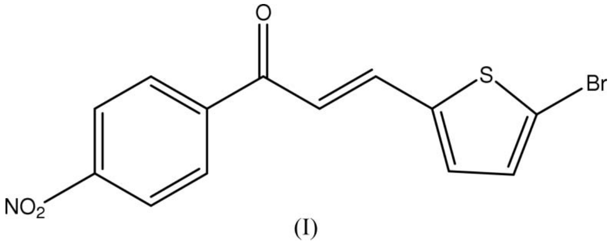
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.004 Å
R factor = 0.041
wR factor = 0.116
Data-to-parameter ratio = 30.7

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

The title compound, $C_{13}H_8BrNO_3S$, crystallizes with two molecules in the asymmetric unit. All bond lengths and angles in both molecules show normal values. In the crystal structure, the molecules are stabilized by intermolecular C—H···O hydrogen bonds and short Br···O interactions.

Comment

The title compound, (I), was prepared as part of our ongoing studies on nonlinear optical (NLO) materials (Patil, Teh *et al.*, 2006*a*,*b*,*c*; Patil, Rosli *et al.*, 2006; Patil, Dharmaprakash *et al.*, 2006; Shettigar *et al.*, 2006; Chantrapromma, Jindawong *et al.*, 2006; Chantrapromma, Ruanwas *et al.*, 2006). Single untwinned crystals of (I) should exhibit second-order nonlinear optical properties as they crystallize in a non-centrosymmetric space group. However, the crystal studied here proved to be subject to partial inversion twinning.



(I)

The asymmetric unit of (I) contains two molecules, *A* and *B* (Fig. 1). The bond lengths and angles in both molecules show normal values (Allen *et al.*, 1987). The dihedral angle between the benzene and the thiophene rings is 25.58 (14)° in molecule *A* and 4.92 (15)° in molecule *B*. The least-squares plane through the enone unit (O1/C7–C9) makes dihedral angles of

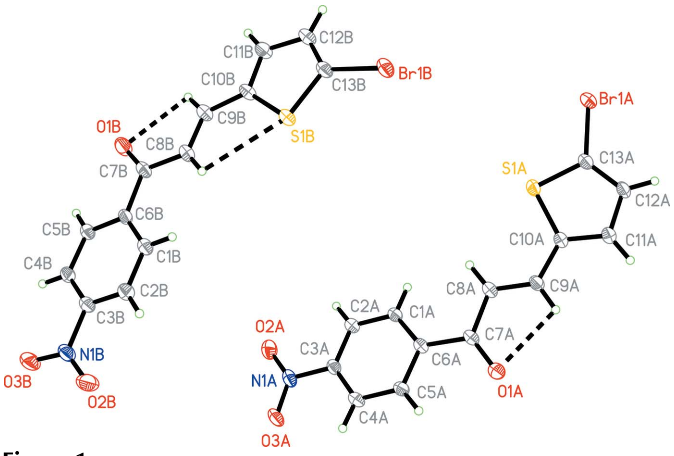


Figure 1
The asymmetric unit of (I), showing 50% probability displacement ellipsoids and the atomic numbering. Hydrogen bonds are shown as dashed lines.

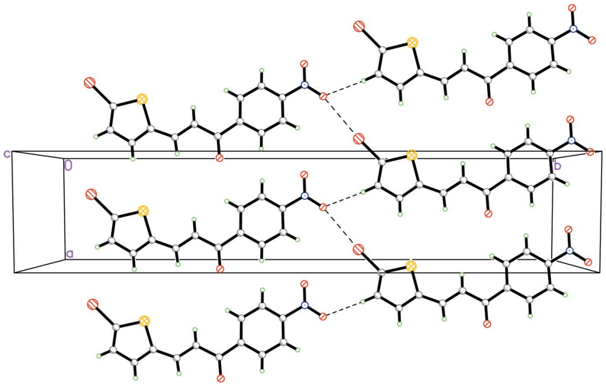


Figure 2
The crystal packing of (I), viewed down the *c* axis. Hydrogen bonds and Br···O short contacts are shown as dashed lines.

18.81 (14) and 7.47 (12)° with the planes of the C1–C6 benzene and C10–C13/S1 thiophene rings, respectively, in molecule *A*, and 4.92 (12) and 1.63 (13)°, respectively, in molecule *B*. The nitro group attached at C3 is almost coplanar with the C1–C6 benzene ring, with O2—N1—C3—C4 torsion angles of 172.4 (3) and 172.9 (3)°, respectively, for *A* and *B*.

Intramolecular C9*A*—H9*AA*···O1*A*, C8*B*—H8*BA*···S1*B* and C9*B*—H9*BA*···O1*B* interactions are observed in the molecular structure of (I). In the crystal structure, the molecules are interconnected by C12*A*—H12*A*···O3*A*$^{\rm iii}$ and C12*B*—H12*B*···O3*B*$^{\rm iv}$ hydrogen bonds (Table 1) and also Br1*A*···O3*A*($-\frac{1}{2}$ + *x*, $\frac{1}{2}$ + *y*, *z*) [3.140 (2) Å] and Br1*B*···O3*B*($\frac{1}{2}$ + *x*, $\frac{1}{2}$ + *y*, *z*) [3.168 (3) Å] short contacts, forming layers parallel to the *ab* plane. These layers are connected by C5*A*—H5*AA*···O1*B*$^{\rm i}$ and C2*B*—H2*BA*···O2*A*$^{\rm ii}$ hydrogen bonds to form a three-dimensional network.

Experimental

5-Bromo-2-thiophenecarbaldehyde (0.01 mol) and 4-nitroacetophenone (0.01 mol) were stirred in 60 ml of methanol at room temperature. A 10% NaOH aqueous solution (5 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 obtained was recrystallized twice from acetone. Crystal suitable for X-ray analysis were grown by slow evaporation of an acetone solution at room temperature.

Crystal data

$C_{13}H_8BrNO_3S$
$M_r = 338.17$
Monoclinic, *Cc*
$a = 6.3062$ (2) Å
$b = 30.3062$ (8) Å
$c = 13.1854$ (4) Å
$\beta = 94.549$ (1)°
$V = 2512.02$ (13) Å^3
$Z = 8$
$D_x = 1.788$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 3.44$ mm^{-1}
$T = 100.0$ (1) K
Block, yellow
0.53 × 0.27 × 0.21 mm

Data collection

Bruker SMART APEX-II CCD
area-detector diffractometer
ω scans
Absorption correction: multi-scan
(*SADABS*; Bruker, 2005)
$T_{\rm min} = 0.260$, $T_{\rm max} = 0.531$
(expected range = 0.238–0.485)
22566 measured reflections
10546 independent reflections
9054 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.046$
$\theta_{\rm max} = 37.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.116$
$S = 1.01$
10546 reflections
344 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0671P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 1.09$ e Å^{-3}
$\Delta\rho_{\rm min} = -1.04$ e Å^{-3}
Absolute structure: Flack (1983),
3980 Friedel pairs
Flack parameter: 0.352 (6)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5*A*—H5*AA*···O1*B*$^{\rm i}$	0.93	2.59	3.284 (4)	132
C9*A*—H9*AA*···O1*A*	0.93	2.44	2.787 (3)	102
C2*B*—H2*BA*···O2*A*$^{\rm ii}$	0.93	2.58	3.313 (4)	136
C8*B*—H8*BA*···S1*B*	0.93	2.88	3.220 (3)	103
C9*B*—H9*BA*···O1*B*	0.93	2.45	2.791 (4)	102
C12*A*—H12*A*···O3*A*$^{\rm iii}$	0.93	2.44	3.362 (4)	171
C12*B*—H12*B*···O3*B*$^{\rm iv}$	0.93	2.47	3.392 (4)	174

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

All H atoms were refined using a riding model, with C—H = 0.93 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$. In the final difference map, the highest peak is located 0.74 Å from atom Br1*B* and the deepest hole is located 1.10 Å from Br1*A*.

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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References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (2005). *APEX2* (Version 1.27), *SAINT* (Version 7.12A) and *SADABS* (Version 2004/1). Bruker AXS Inc., Madison, Wisconsin, USA.
Chantrapromma, S., Jindawong, B., Fun, H.-K., Patil, P. S. & Karalai, C. (2006). *Acta Cryst.* E**62**, o1802–o1804.
Chantrapromma, S., Ruanwas, P., Fun, H.-K. & Patil, P. S. (2006). *Acta Cryst.* E**62**, o5494–o5496.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Patil, P. S., Dharmaprakash, S. M., Fun, H.-K. & Karthikeyan, M. S. (2006). *J. Cryst. Growth*, **297**, 111–116.
Patil, P. S., Rosli, M. M., Fun, H.-K., Razak, I. A., Puranik, V. G. & Dharmaprakash, S. M. (2006). *Acta Cryst.* E**62**, o4798–o4799.
Patil, P. S., Teh, J. B.-J., Fun, H.-K., Razak, I. A. & Dharmaprakash, S. M. (2006*a*). *Acta Cryst.* E**62**, o896–o898.
Patil, P. S., Teh, J. B.-J., Fun, H.-K., Razak, I. A. & Dharmaprakash, S. M. (2006*b*). *Acta Cryst.* E**62**, o1710–o1712.
Patil, P. S., Teh, J. B.-J., Fun, H.-K., Razak, I. A. & Dharmaprakash, S. M. (2006*c*). *Acta Cryst.* E**62**, o3096–o3098.
Sheldrick, G. M. (1998). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Shettigar, V., Patil, P. S., Naveen, S., Dharmaprakash, S. M., Sridhar, M. A. & Shashidra Prasad, J. (2006). *J. Cryst. Growth*, **295**, 44–49.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

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