

(2E)-1-(3-Bromothien-2-yl)-3-phenylprop-2-en-1-oneRay J. Butcher,^a Jerry P. Jasinski,^{b*} H. S. Yathirajan,^c B. V. Ashalatha^d and B. Narayana^d^aDepartment of Chemistry, Howard University, 525 College Street NW, Washington, DC 20059, USA, ^bDepartment of Chemistry, Keene State College, 229 Main Street, Keene, NH 03435-2001, USA, ^cDepartment of Studies in Chemistry, University of Mysore, Manasagangotri, Mysore 570 006, India, and ^dDepartment of Chemistry, Mangalore University, Mangalagangotri 574 199, India
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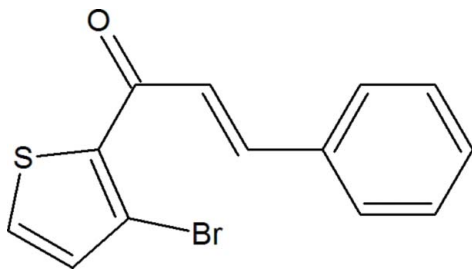
Received 23 July 2007; accepted 1 August 2007

Key indicators: single-crystal X-ray study; $T = 123$ K; mean $\sigma(\text{C}-\text{C}) = 0.011$ Å; R factor = 0.069; wR factor = 0.178; data-to-parameter ratio = 19.3.

The title compound, $\text{C}_{13}\text{H}_9\text{BrOS}$, crystallizes with two independent molecules (*A* and *B*) in the asymmetric unit. The mean planes of the 3-bromothien-2-yl and 3-phenyl groups in *A* and *B* form dihedral angles of 4.9 (7) and 12.2 (4)°, respectively. The angles between the mean plane of the prop-2-en-1-one group and those of the 3-bromothien-2-yl and 3-phenyl groups are 2.8 (2) and 3.8 (2)°, respectively, in molecule *A*, and 5.1 (1) and 9.8 (9)° in molecule *B*. Essentially planar groups of molecule *A* pack zigzag to similar groups of molecule *B* along the *a* axis of the unit cell.

Related literature

For related structures, see: Baxter *et al.* (1990); Ng *et al.* (2006); Yathirajan, Sarojini, Narayana, Ashalatha & Bolte (2006); Yathirajan, Sarojini, Narayana, Bindya & Bolte (2006); Harrison *et al.* (2006); Butcher *et al.* (2007*a,b,c*). For related background, see: Fichou *et al.* (1988); Goto *et al.* (1991); Cho *et al.* (1996); Uchida *et al.* (1998); Tam *et al.* (1989); Indira *et al.* (2002); Opletalova & Sedivy (1999); Butcher, Yathirajan, Sarojini *et al.* (2006); Butcher *et al.* (2006*a,b*).

**Experimental***Crystal data* $\text{C}_{13}\text{H}_9\text{BrOS}$
 $M_r = 293.17$
Orthorhombic, $Pca2_1$
 $a = 17.321$ (4) Å
 $b = 5.4295$ (12) Å
 $c = 24.600$ (5) Å
 $V = 2313.5$ (9) Å³
 $Z = 8$
Mo $K\alpha$ radiation
 $\mu = 3.71$ mm⁻¹
 $T = 123$ K
 $0.69 \times 0.32 \times 0.10$ mm*Data collection*Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.335$, $T_{\max} = 1.000$
(expected range = 0.236–0.703)
17031 measured reflections
5590 independent reflections
4041 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.105$ *Refinement* $R[F^2 > 2\sigma(F^2)] = 0.069$
 $wR(F^2) = 0.178$
 $S = 0.99$
5590 reflections
289 parameters
1 restraint
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 2.41$ e Å⁻³
 $\Delta\rho_{\text{min}} = -1.49$ e Å⁻³
Absolute structure: Flack (1983), with 2580 Friedel pairs
Flack parameter: 0.014 (15)

Data collection: APEX2 (Bruker, 2006); cell refinement: APEX2; data reduction: APEX2; program(s) used to solve structure: SHELXS97 (Sheldrick, 1990); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXTL (Bruker, 2000).

BVA thanks Mangalore University for access to their research facilities. RJB acknowledges the Laboratory for the Structure of Matter at the Naval Research Laboratory for access to their diffractometers.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2454).

References

- Baxter, L. A. M., Blake, A. J., Heath, G. A. & Stephenson, T. A. (1990). *Acta Cryst.* **C46**, 508–510.
Bruker (2000). SHELXTL. Version 6.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2006). APEX2. Version 2.1-0. Bruker AXS Inc., Madison, Wisconsin, USA.
Butcher, R. J., Yathirajan, H. S., Anilkumar, H. G., Sarojini, B. K. & Narayana, B. (2006*a*). *Acta Cryst.* **E62**, o1633–o1635.
Butcher, R. J., Yathirajan, H. S., Anilkumar, H. G., Sarojini, B. K. & Narayana, B. (2006*b*). *Acta Cryst.* **E62**, o1659–o1661.
Butcher, R. J., Yathirajan, H. S., Ashalatha, B. V., Narayana, B. & Sarojini, B. K. (2007*a*). *Acta Cryst.* **E63**, o1005–o1007.
Butcher, R. J., Yathirajan, H. S., Ashalatha, B. V., Narayana, B. & Sarojini, B. K. (2007*b*). *Acta Cryst.* **E63**, o1201–o1203.
Butcher, R. J., Yathirajan, H. S., Ashalatha, B. V., Narayana, B. & Sarojini, B. K. (2007*c*). *Acta Cryst.* **E63**, o1430–o1431.
Butcher, R. J., Yathirajan, H. S., Sarojini, B. K., Narayana, B. & Mithun, A. (2006). *Acta Cryst.* **E62**, o1629–o1630.
Cho, B. R., Je, J. T., Kim, H. S., Jeon, S. J., Song, O. K. & Wang, C. H. (1996). *Bull. Korean Chem. Soc.* **17**, 693–695.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Fichou, D., Watanabe, T., Takeda, T., Miyata, S., Goto, Y. & Nakayama, M. (1988). *Jpn. J. Appl. Phys.* **27**, L429–L430.
Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.

- Goto, Y., Hayashi, A., Kimura, Y. & Nakayama, M. (1991). *J. Cryst. Growth*, **108**, 688–698.
- Harrison, W. T. A., Yathirajan, H. S., Ashalatha, B. V., Bindya, S. & Narayana, B. (2006). *Acta Cryst. E***62**, o4164–o4165.
- Indira, J., Karat, P. P. & Sarojini, B. K. (2002). *J. Cryst. Growth*, **242**, 209–214.
- Ng, S.-L., Patil, P. S., Razak, I. A., Fun, H.-K. & Dharmaparakash, S. M. (2006). *Acta Cryst. E***62**, o3200–o3202.
- Opletalova, V. & Sedivy, D. (1999). *Ceska Slov. Farm.* **48**, 252–255.
- Sheldrick, G. M. (1990). *Acta Cryst. A***46**, 467–473.
- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
- Tam, W., Guerin, B., Calabrese, J. C. & Stevenson, S. H. (1989). *Chem. Phys. Lett.* **154**, 93–96.
- Uchida, T., Kozawa, K., Sakai, T., Aoki, M., Yoguchi, H., Abduryim, A. & Watanabe, Y. (1998). *Mol. Cryst. Liq. Cryst.* **315**, 135–140.
- Yathirajan, H. S., Sarojini, B. K., Narayana, B., Ashalatha, B. V. & Bolte, M. (2006). *Acta Cryst. E***62**, o3964–o3965.
- Yathirajan, H. S., Sarojini, B. K., Narayana, B., Bindya, S. & Bolte, M. (2006). *Acta Cryst. E***62**, o4048–o4049.

supplementary materials

Acta Cryst. (2007). E63, o3705-o3706 [doi:10.1107/S1600536807037841]

(2E)-1-(3-Bromothien-2-yl)-3-phenylprop-2-en-1-one

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Comment

Chalcones and their heterocyclic derivatives show numerous biological effects. Among several organic compounds reported for non-linear optical (NLO) property, chalcone derivatives are noticeable materials for their excellent blue light transmittance and good crystallizability. Chalcones provide a necessary configuration to show NLO property with two planar rings connected through a conjugated double bond. The NLO effect in organic molecules originates from a strong intermolecular donor-acceptor interaction, a delocalized π -electron system, and the ability to crystallize in a non-centrosymmetric structure. Secondly, the backbone is usually twisted, and this twist is inherently chiral and often results in these compounds crystallizing in non-centrosymmetric space groups. Substitution on either of the phenyl rings greatly influences non-centrosymmetric crystal packing. It is speculated that, in order to improve the activity, more bulky substituents should be introduced to increase the spontaneous polarization of a non-centrosymmetric crystal structure. The molecular hyperpolarizability, β , is strongly influenced not only by the electronic effect, but also by the steric effect of the substituent. Prompted by this, and in continuation of our quest to synthesize new materials which can find use in the photonics industries, we have synthesized a new chalcone and the present paper reports the crystal structure of a newly synthesized chalcone, (I), $C_{13}H_9BrOS$.

The mean planes of the 3-Bromothien-2-yl and 3-phenyl groups in molecules A and B form dihedral angles of $4.9 (7)^\circ$ and $12.2 (4)^\circ$, respectively, with each other (Fig. 1). The angles between the mean plane of the prop-2-en-1-one group and those of the 3-Bromothien-2-yl and 3-phenyl groups are $2.8 (2)^\circ$ and $3.8 (2)^\circ$ in molecule A, and $5.1 (1)^\circ$ and $9.8 (9)^\circ$ in molecule B.

The packing diagram displays a zigzag array of mean planes of adjacent planar arranged groups of molecules A located next to mean planes of molecules B located diagonal along the a axis of the unit cell (Fig. 2). The closest centroid-centroid distance of $4.63 (2) \text{ \AA}$ occurs between the nearby mean planes of the inverted 3-Bromothien-2-yl and 3-phenyl groups for molecule A.

Experimental

3-bromo-2-acetylthiophene (10 g, 0.048 mol) in 50 ml methanol is mixed with benzaldehyde (5.0 g, 0.048 mol) and the mixture was treated with an 10 ml of 30% potassium hydroxide solution at 278 K (Fig. 3). The reaction mixture was then brought to room temperature and stirred for 3 h. The solid precipitated was filtered and washed with water, dried and recrystallized from toluene (m.p.: 339 K).

Refinement

The high value of R_{int} is probably due to poor crystal quality. The H atoms were included in the riding model approximation with $C-H = 0.95 \text{ \AA}$, and with $U_{\text{iso}}(H) = 1.18-1.21 U_{\text{eq}}(C)$. The maximum residual electron density peaks of 2.40 and $-1.49 e \text{ \AA}^{-3}$, were located at 0.90 and 0.76 \AA from the Br1A and Br1B atoms, respectively.

Figures

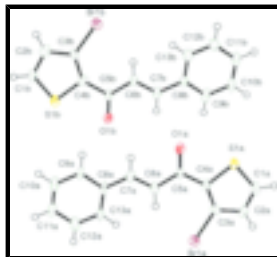


Fig. 1. Molecular structure of $C_{13}H_9BrOS$, (I), showing atom labeling and 50% probability displacement ellipsoids for independent molecules A and B in the asymmetric unit.

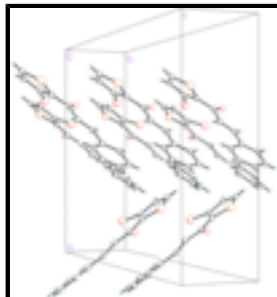


Fig. 2. Packing diagram of $C_{13}H_9BrOS$ viewed down the b axis.

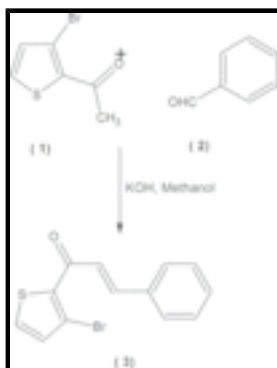


Fig. 3. Synthesis scheme of $C_{13}H_9BrOS$.

(2E)-1-(3-Bromothiien-2-yl)-3-phenylprop-2-en-1-one

Crystal data

$C_{13}H_9BrOS$

$M_r = 293.17$

Orthorhombic, $Pca2_1$

$a = 17.321$ (4) Å

$b = 5.4295$ (12) Å

$c = 24.600$ (5) Å

$V = 2313.5$ (9) Å³

$Z = 8$

$F_{000} = 1168$

$D_x = 1.683$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 5433 reflections

$\theta = 2.4$ – 28.3°

$\mu = 3.71$ mm⁻¹

$T = 123$ K

Rectangular, colourless

$0.69 \times 0.32 \times 0.10$ mm

Data collection

| | |
|---|--|
| Bruker APEXII CCD area-detector diffractometer | 5590 independent reflections |
| Radiation source: fine-focus sealed tube | 4041 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{\text{int}} = 0.105$ |
| $T = 123$ K | $\theta_{\text{max}} = 28.7^\circ$ |
| φ and ω scans | $\theta_{\text{min}} = 2.4^\circ$ |
| Absorption correction: multi-scan (SADABS; Sheldrick, 1996) | $h = -23 \rightarrow 23$ |
| $T_{\text{min}} = 0.335$, $T_{\text{max}} = 1.000$ | $k = -5 \rightarrow 7$ |
| 17031 measured reflections | $l = -31 \rightarrow 33$ |

Refinement

| | |
|--|--|
| Refinement on F^2 | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | H-atom parameters constrained |
| $R[F^2 > 2\sigma(F^2)] = 0.069$ | $w = 1/[\sigma^2(F_o^2) + (0.114P)^2]$ |
| $wR(F^2) = 0.178$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 0.99$ | $(\Delta/\sigma)_{\text{max}} = 0.001$ |
| 5590 reflections | $\Delta\rho_{\text{max}} = 2.41 \text{ e } \text{\AA}^{-3}$ |
| 289 parameters | $\Delta\rho_{\text{min}} = -1.49 \text{ e } \text{\AA}^{-3}$ |
| 1 restraint | Extinction correction: none |
| Primary atom site location: structure-invariant direct methods | Absolute structure: Flack (1983), 2580 Friedel pairs |
| Secondary atom site location: difference Fourier map | Flack parameter: 0.014 (15) |

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|---------------|-------------|----------------------------------|
| Br1A | 0.19487 (4) | -0.04060 (15) | 0.04400 (3) | 0.0344 (2) |
| Br1B | -0.10871 (5) | 0.54938 (16) | 0.45589 (3) | 0.0362 (2) |
| S1A | 0.25796 (9) | -0.2937 (3) | 0.20837 (8) | 0.0247 (4) |

supplementary materials

| | | | | |
|------|--------------|--------------|-------------|-------------|
| S1B | -0.15628 (9) | 0.7896 (3) | 0.28805 (8) | 0.0242 (4) |
| O1A | 0.1645 (3) | 0.1132 (10) | 0.2363 (2) | 0.0248 (10) |
| O1B | -0.0611 (3) | 0.3863 (11) | 0.2652 (2) | 0.0309 (12) |
| C1A | 0.2977 (4) | -0.4717 (15) | 0.1588 (4) | 0.0274 (17) |
| H1AA | 0.3305 | -0.6085 | 0.1656 | 0.033* |
| C2A | 0.2771 (4) | -0.3951 (16) | 0.1081 (3) | 0.0291 (17) |
| H2AA | 0.2935 | -0.4729 | 0.0755 | 0.035* |
| C3A | 0.2279 (4) | -0.1846 (13) | 0.1098 (3) | 0.0218 (14) |
| C4A | 0.2109 (4) | -0.1080 (15) | 0.1615 (3) | 0.0224 (15) |
| C5A | 0.1631 (3) | 0.0939 (14) | 0.1870 (3) | 0.0220 (15) |
| C6A | 0.1159 (3) | 0.2476 (14) | 0.1517 (3) | 0.0233 (15) |
| H6AA | 0.1161 | 0.2209 | 0.1135 | 0.028* |
| C7A | 0.0719 (4) | 0.4273 (14) | 0.1731 (3) | 0.0258 (16) |
| H7AA | 0.0741 | 0.4479 | 0.2114 | 0.031* |
| C8A | 0.0203 (4) | 0.5969 (14) | 0.1426 (3) | 0.0234 (15) |
| C9A | -0.0133 (3) | 0.7948 (14) | 0.1707 (4) | 0.0282 (16) |
| H9AA | -0.0033 | 0.8184 | 0.2083 | 0.034* |
| C10A | -0.0618 (4) | 0.9568 (14) | 0.1427 (4) | 0.0323 (19) |
| H10A | -0.0856 | 1.0885 | 0.1617 | 0.039* |
| C11A | -0.0754 (4) | 0.9286 (14) | 0.0882 (4) | 0.0329 (19) |
| H11A | -0.1064 | 1.0446 | 0.0693 | 0.040* |
| C12A | -0.0432 (4) | 0.7272 (16) | 0.0604 (3) | 0.0350 (19) |
| H12A | -0.0541 | 0.7031 | 0.0229 | 0.042* |
| C13A | 0.0046 (4) | 0.5632 (16) | 0.0877 (3) | 0.0321 (18) |
| H13A | 0.0267 | 0.4278 | 0.0688 | 0.039* |
| C1B | -0.1999 (4) | 0.9667 (14) | 0.3363 (4) | 0.0276 (16) |
| H1BA | -0.2330 | 1.1005 | 0.3276 | 0.033* |
| C2B | -0.1839 (4) | 0.9028 (15) | 0.3871 (3) | 0.0253 (16) |
| H2BA | -0.2026 | 0.9852 | 0.4185 | 0.030* |
| C3B | -0.1345 (4) | 0.6920 (14) | 0.3877 (3) | 0.0243 (14) |
| C4B | -0.1132 (3) | 0.6062 (15) | 0.3372 (3) | 0.0220 (15) |
| C5B | -0.0641 (4) | 0.4081 (12) | 0.3151 (3) | 0.0206 (14) |
| C6B | -0.0212 (4) | 0.2400 (15) | 0.3516 (3) | 0.0272 (16) |
| H6BA | -0.0264 | 0.2568 | 0.3899 | 0.033* |
| C7B | 0.0246 (4) | 0.0655 (14) | 0.3314 (3) | 0.0250 (15) |
| H7BA | 0.0260 | 0.0508 | 0.2929 | 0.030* |
| C8B | 0.0732 (3) | -0.1073 (13) | 0.3625 (3) | 0.0252 (16) |
| C9B | 0.1140 (4) | -0.2874 (13) | 0.3332 (3) | 0.0254 (15) |
| H9BA | 0.1109 | -0.2924 | 0.2946 | 0.030* |
| C10B | 0.1595 (4) | -0.4607 (15) | 0.3615 (4) | 0.0330 (18) |
| H10B | 0.1873 | -0.5822 | 0.3417 | 0.040* |
| C11B | 0.1642 (4) | -0.4563 (15) | 0.4168 (4) | 0.0354 (19) |
| H11B | 0.1947 | -0.5741 | 0.4356 | 0.042* |
| C12B | 0.1238 (5) | -0.2768 (18) | 0.4451 (4) | 0.046 (2) |
| H12B | 0.1277 | -0.2700 | 0.4836 | 0.055* |
| C13B | 0.0778 (5) | -0.1073 (16) | 0.4182 (4) | 0.0320 (17) |
| H13B | 0.0492 | 0.0102 | 0.4385 | 0.038* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|------|------------|------------|------------|------------|-------------|-------------|
| Br1A | 0.0368 (4) | 0.0379 (5) | 0.0284 (4) | 0.0097 (3) | -0.0013 (3) | 0.0006 (4) |
| Br1B | 0.0410 (4) | 0.0391 (5) | 0.0286 (4) | 0.0139 (3) | 0.0015 (3) | 0.0031 (5) |
| S1A | 0.0172 (7) | 0.0230 (9) | 0.0340 (9) | 0.0013 (6) | -0.0024 (6) | 0.0024 (8) |
| S1B | 0.0190 (7) | 0.0232 (9) | 0.0303 (9) | 0.0022 (6) | -0.0020 (6) | -0.0003 (7) |
| O1A | 0.020 (2) | 0.022 (3) | 0.032 (3) | 0.003 (2) | 0.003 (2) | 0.001 (2) |
| O1B | 0.031 (3) | 0.027 (3) | 0.034 (3) | 0.011 (2) | 0.004 (2) | 0.001 (2) |
| C1A | 0.009 (3) | 0.028 (4) | 0.045 (5) | 0.005 (3) | -0.002 (3) | -0.002 (3) |
| C2A | 0.017 (3) | 0.033 (5) | 0.037 (4) | -0.005 (3) | 0.000 (3) | -0.004 (3) |
| C3A | 0.021 (3) | 0.017 (4) | 0.027 (4) | 0.000 (2) | -0.003 (3) | 0.006 (3) |
| C4A | 0.018 (3) | 0.021 (4) | 0.029 (4) | -0.008 (3) | 0.002 (3) | -0.008 (3) |
| C5A | 0.007 (2) | 0.026 (4) | 0.034 (4) | 0.001 (2) | 0.001 (2) | 0.002 (3) |
| C6A | 0.017 (3) | 0.021 (4) | 0.031 (4) | 0.003 (3) | 0.000 (3) | -0.001 (3) |
| C7A | 0.016 (3) | 0.022 (4) | 0.040 (4) | 0.001 (2) | -0.002 (3) | 0.001 (3) |
| C8A | 0.013 (3) | 0.022 (4) | 0.035 (4) | 0.000 (2) | 0.002 (3) | 0.003 (3) |
| C9A | 0.012 (3) | 0.021 (4) | 0.051 (5) | -0.004 (3) | 0.002 (3) | -0.001 (3) |
| C10A | 0.014 (3) | 0.020 (4) | 0.063 (6) | 0.003 (3) | 0.007 (3) | 0.011 (4) |
| C11A | 0.019 (3) | 0.020 (4) | 0.060 (6) | 0.001 (3) | 0.000 (3) | 0.014 (4) |
| C12A | 0.029 (3) | 0.038 (5) | 0.038 (5) | 0.005 (3) | 0.000 (3) | 0.011 (3) |
| C13A | 0.027 (3) | 0.030 (5) | 0.040 (5) | 0.004 (3) | 0.003 (3) | 0.004 (4) |
| C1B | 0.018 (3) | 0.020 (4) | 0.045 (5) | 0.000 (3) | -0.002 (3) | -0.001 (3) |
| C2B | 0.015 (3) | 0.025 (4) | 0.035 (4) | -0.005 (3) | 0.007 (3) | -0.006 (3) |
| C3B | 0.023 (3) | 0.021 (4) | 0.028 (4) | -0.007 (3) | -0.003 (3) | 0.003 (3) |
| C4B | 0.008 (2) | 0.022 (4) | 0.036 (4) | 0.000 (2) | 0.002 (2) | 0.006 (3) |
| C5B | 0.021 (3) | 0.007 (3) | 0.034 (4) | -0.002 (2) | 0.000 (3) | -0.003 (3) |
| C6B | 0.020 (3) | 0.030 (4) | 0.032 (4) | 0.005 (3) | -0.005 (3) | 0.000 (3) |
| C7B | 0.014 (3) | 0.025 (4) | 0.036 (4) | 0.001 (3) | -0.002 (3) | -0.001 (3) |
| C8B | 0.012 (3) | 0.011 (3) | 0.053 (5) | 0.003 (2) | -0.003 (3) | -0.004 (3) |
| C9B | 0.018 (3) | 0.012 (3) | 0.046 (4) | 0.000 (2) | 0.004 (3) | -0.001 (3) |
| C10B | 0.014 (3) | 0.025 (4) | 0.060 (6) | 0.003 (3) | 0.001 (3) | 0.001 (4) |
| C11B | 0.020 (3) | 0.030 (5) | 0.056 (6) | 0.009 (3) | -0.003 (3) | 0.004 (4) |
| C12B | 0.050 (5) | 0.038 (5) | 0.050 (6) | 0.019 (4) | -0.009 (4) | 0.006 (4) |
| C13B | 0.033 (4) | 0.023 (4) | 0.039 (4) | 0.015 (3) | -0.009 (3) | -0.008 (3) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|----------|------------|-----------|------------|
| Br1A—C3A | 1.886 (7) | C11A—H11A | 0.9500 |
| Br1B—C3B | 1.900 (7) | C12A—C13A | 1.389 (11) |
| S1A—C1A | 1.702 (8) | C12A—H12A | 0.9500 |
| S1A—C4A | 1.736 (8) | C13A—H13A | 0.9500 |
| S1B—C1B | 1.705 (8) | C1B—C2B | 1.324 (12) |
| S1B—C4B | 1.735 (8) | C1B—H1BA | 0.9500 |
| O1A—C5A | 1.216 (9) | C2B—C3B | 1.429 (11) |
| O1B—C5B | 1.235 (9) | C2B—H2BA | 0.9500 |
| C1A—C2A | 1.361 (12) | C3B—C4B | 1.378 (11) |
| C1A—H1AA | 0.9500 | C4B—C5B | 1.475 (10) |

supplementary materials

| | | | |
|----------------|------------|----------------|------------|
| C2A—C3A | 1.426 (10) | C5B—C6B | 1.480 (10) |
| C2A—H2AA | 0.9500 | C6B—C7B | 1.332 (10) |
| C3A—C4A | 1.371 (10) | C6B—H6BA | 0.9500 |
| C4A—C5A | 1.510 (10) | C7B—C8B | 1.476 (10) |
| C5A—C6A | 1.457 (10) | C7B—H7BA | 0.9500 |
| C6A—C7A | 1.345 (10) | C8B—C13B | 1.371 (12) |
| C6A—H6AA | 0.9500 | C8B—C9B | 1.406 (10) |
| C7A—C8A | 1.487 (10) | C9B—C10B | 1.412 (11) |
| C7A—H7AA | 0.9500 | C9B—H9BA | 0.9500 |
| C8A—C13A | 1.390 (11) | C10B—C11B | 1.363 (13) |
| C8A—C9A | 1.403 (10) | C10B—H10B | 0.9500 |
| C9A—C10A | 1.397 (11) | C11B—C12B | 1.388 (12) |
| C9A—H9AA | 0.9500 | C11B—H11B | 0.9500 |
| C10A—C11A | 1.370 (13) | C12B—C13B | 1.386 (11) |
| C10A—H10A | 0.9500 | C12B—H12B | 0.9500 |
| C11A—C12A | 1.405 (12) | C13B—H13B | 0.9500 |
| C1A—S1A—C4A | 92.5 (4) | C8A—C13A—H13A | 119.9 |
| C1B—S1B—C4B | 91.7 (4) | C2B—C1B—S1B | 114.6 (6) |
| C2A—C1A—S1A | 112.1 (6) | C2B—C1B—H1BA | 122.7 |
| C2A—C1A—H1AA | 123.9 | S1B—C1B—H1BA | 122.7 |
| S1A—C1A—H1AA | 123.9 | C1B—C2B—C3B | 110.2 (7) |
| C1A—C2A—C3A | 112.0 (7) | C1B—C2B—H2BA | 124.9 |
| C1A—C2A—H2AA | 124.0 | C3B—C2B—H2BA | 124.9 |
| C3A—C2A—H2AA | 124.0 | C4B—C3B—C2B | 114.9 (7) |
| C4A—C3A—C2A | 113.5 (6) | C4B—C3B—Br1B | 126.5 (6) |
| C4A—C3A—Br1A | 127.2 (6) | C2B—C3B—Br1B | 118.5 (6) |
| C2A—C3A—Br1A | 119.3 (5) | C3B—C4B—C5B | 137.1 (7) |
| C3A—C4A—C5A | 136.4 (7) | C3B—C4B—S1B | 108.6 (6) |
| C3A—C4A—S1A | 109.8 (6) | C5B—C4B—S1B | 114.2 (6) |
| C5A—C4A—S1A | 113.7 (5) | O1B—C5B—C4B | 117.4 (6) |
| O1A—C5A—C6A | 123.8 (6) | O1B—C5B—C6B | 121.5 (6) |
| O1A—C5A—C4A | 117.8 (6) | C4B—C5B—C6B | 121.1 (6) |
| C6A—C5A—C4A | 118.3 (7) | C7B—C6B—C5B | 120.7 (7) |
| C7A—C6A—C5A | 120.0 (7) | C7B—C6B—H6BA | 119.6 |
| C7A—C6A—H6AA | 120.0 | C5B—C6B—H6BA | 119.6 |
| C5A—C6A—H6AA | 120.0 | C6B—C7B—C8B | 126.8 (8) |
| C6A—C7A—C8A | 126.4 (7) | C6B—C7B—H7BA | 116.6 |
| C6A—C7A—H7AA | 116.8 | C8B—C7B—H7BA | 116.6 |
| C8A—C7A—H7AA | 116.8 | C13B—C8B—C9B | 118.9 (7) |
| C13A—C8A—C9A | 119.9 (7) | C13B—C8B—C7B | 123.5 (7) |
| C13A—C8A—C7A | 121.7 (7) | C9B—C8B—C7B | 117.5 (7) |
| C9A—C8A—C7A | 118.4 (7) | C8B—C9B—C10B | 119.4 (8) |
| C10A—C9A—C8A | 119.2 (8) | C8B—C9B—H9BA | 120.3 |
| C10A—C9A—H9AA | 120.4 | C10B—C9B—H9BA | 120.3 |
| C8A—C9A—H9AA | 120.4 | C11B—C10B—C9B | 120.9 (7) |
| C11A—C10A—C9A | 121.1 (8) | C11B—C10B—H10B | 119.5 |
| C11A—C10A—H10A | 119.5 | C9B—C10B—H10B | 119.5 |
| C9A—C10A—H10A | 119.5 | C10B—C11B—C12B | 118.9 (8) |
| C10A—C11A—C12A | 119.6 (7) | C10B—C11B—H11B | 120.5 |

| | | | |
|---------------------|------------|---------------------|------------|
| C10A—C11A—H11A | 120.2 | C12B—C11B—H11B | 120.5 |
| C12A—C11A—H11A | 120.2 | C13B—C12B—C11B | 121.1 (9) |
| C13A—C12A—C11A | 120.0 (8) | C13B—C12B—H12B | 119.5 |
| C13A—C12A—H12A | 120.0 | C11B—C12B—H12B | 119.5 |
| C11A—C12A—H12A | 120.0 | C8B—C13B—C12B | 120.7 (8) |
| C12A—C13A—C8A | 120.1 (8) | C8B—C13B—H13B | 119.6 |
| C12A—C13A—H13A | 119.9 | C12B—C13B—H13B | 119.6 |
| C4A—S1A—C1A—C2A | 0.3 (6) | C4B—S1B—C1B—C2B | 1.1 (6) |
| S1A—C1A—C2A—C3A | 0.4 (8) | S1B—C1B—C2B—C3B | -1.6 (8) |
| C1A—C2A—C3A—C4A | -1.2 (9) | C1B—C2B—C3B—C4B | 1.4 (9) |
| C1A—C2A—C3A—Br1A | 178.0 (5) | C1B—C2B—C3B—Br1B | -175.8 (5) |
| C2A—C3A—C4A—C5A | -179.2 (7) | C2B—C3B—C4B—C5B | 178.3 (7) |
| Br1A—C3A—C4A—C5A | 1.6 (12) | Br1B—C3B—C4B—C5B | -4.8 (12) |
| C2A—C3A—C4A—S1A | 1.4 (7) | C2B—C3B—C4B—S1B | -0.6 (7) |
| Br1A—C3A—C4A—S1A | -177.7 (4) | Br1B—C3B—C4B—S1B | 176.3 (4) |
| C1A—S1A—C4A—C3A | -1.0 (5) | C1B—S1B—C4B—C3B | -0.2 (5) |
| C1A—S1A—C4A—C5A | 179.5 (5) | C1B—S1B—C4B—C5B | -179.4 (5) |
| C3A—C4A—C5A—O1A | -176.5 (7) | C3B—C4B—C5B—O1B | 178.2 (8) |
| S1A—C4A—C5A—O1A | 2.8 (8) | S1B—C4B—C5B—O1B | -2.9 (8) |
| C3A—C4A—C5A—C6A | 5.9 (12) | C3B—C4B—C5B—C6B | -0.8 (12) |
| S1A—C4A—C5A—C6A | -174.8 (5) | S1B—C4B—C5B—C6B | 178.1 (5) |
| O1A—C5A—C6A—C7A | 1.8 (11) | O1B—C5B—C6B—C7B | 2.7 (11) |
| C4A—C5A—C6A—C7A | 179.2 (6) | C4B—C5B—C6B—C7B | -178.3 (7) |
| C5A—C6A—C7A—C8A | -179.3 (6) | C5B—C6B—C7B—C8B | 177.1 (7) |
| C6A—C7A—C8A—C13A | 8.7 (11) | C6B—C7B—C8B—C13B | -0.6 (12) |
| C6A—C7A—C8A—C9A | -171.5 (7) | C6B—C7B—C8B—C9B | 176.4 (7) |
| C13A—C8A—C9A—C10A | -0.5 (10) | C13B—C8B—C9B—C10B | -1.0 (10) |
| C7A—C8A—C9A—C10A | 179.7 (6) | C7B—C8B—C9B—C10B | -178.2 (6) |
| C8A—C9A—C10A—C11A | -1.6 (10) | C8B—C9B—C10B—C11B | 0.2 (11) |
| C9A—C10A—C11A—C12A | 3.0 (11) | C9B—C10B—C11B—C12B | -0.4 (12) |
| C10A—C11A—C12A—C13A | -2.5 (11) | C10B—C11B—C12B—C13B | 1.4 (13) |
| C11A—C12A—C13A—C8A | 0.4 (12) | C9B—C8B—C13B—C12B | 2.1 (12) |
| C9A—C8A—C13A—C12A | 1.0 (11) | C7B—C8B—C13B—C12B | 179.1 (8) |
| C7A—C8A—C13A—C12A | -179.2 (7) | C11B—C12B—C13B—C8B | -2.3 (14) |

Fig. 1

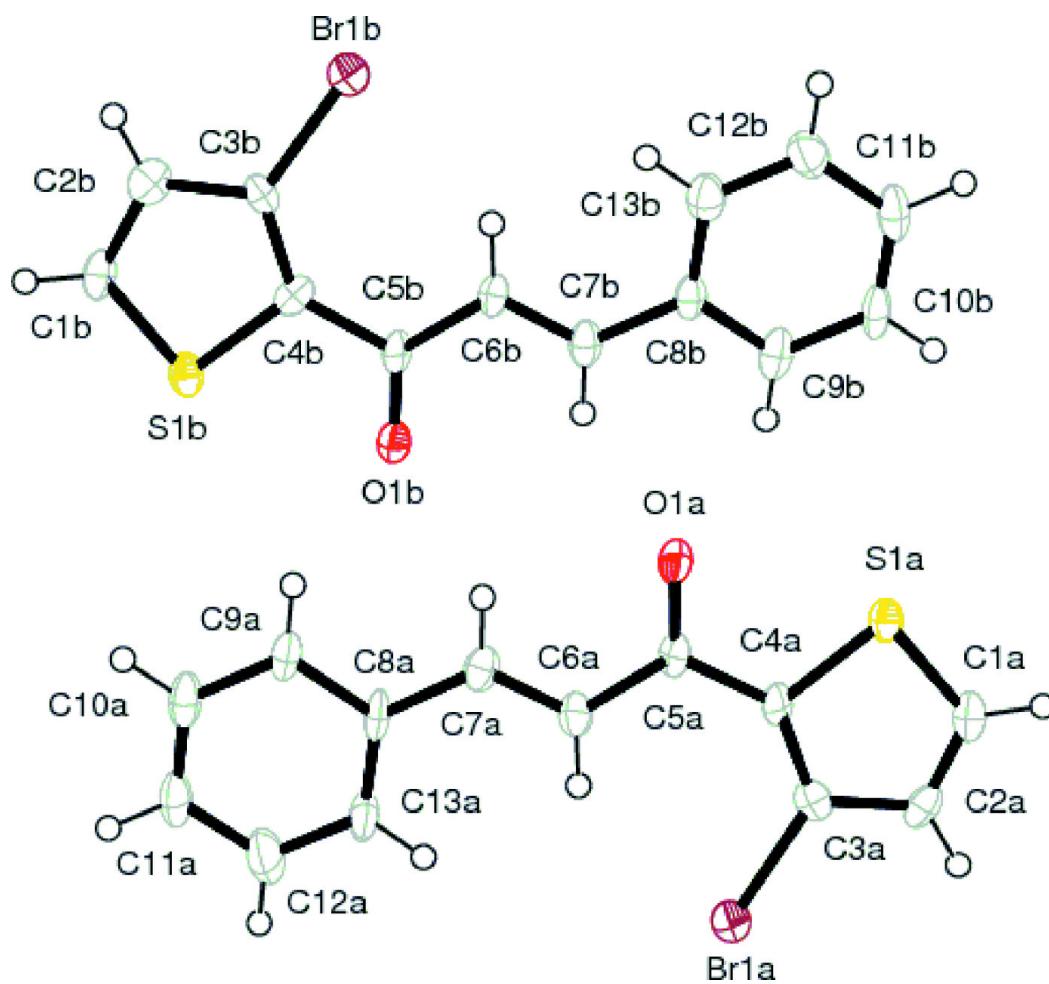


Fig. 2

