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

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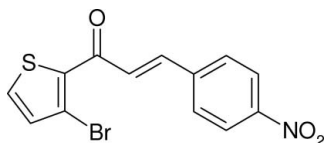Correspondence e-mail: w.harrison@abdn.ac.uk

In the title compound, $C_{13}H_8BrNO_3S$, the dihedral angles between the enone fragment and the adjacent thienyl (th) and benzene (bz) rings are 12.7 (2) and 2.8 (2)°, respectively. The dihedral angle between the th and bz ring systems is 13.20 (18)°. An acute C—H⋯O interaction may help to consolidate the centrosymmetric crystal packing.

Related literature

For related structures and background, see: Harrison *et al.* (2007). For reference structural data, see: Allen *et al.* (1987). For general background, see: Uchida *et al.* (1998).

Experimental

Crystal data

$C_{13}H_8BrNO_3S$	$a = 4.8098$ (3) Å
$M_r = 338.17$	$b = 10.7863$ (8) Å
Triclinic, $P\bar{1}$	$c = 12.7709$ (9) Å
$\alpha = 81.835$ (1)°	Mo $K\alpha$ radiation
$\beta = 83.714$ (2)°	$\mu = 3.32$ mm^{-1}
$\gamma = 85.316$ (2)°	$T = 291$ (2) K
$V = 650.42$ (8) Å^3	$0.47 \times 0.20 \times 0.20$ mm
$Z = 2$	

Data collection

Bruker SMART1000 CCD diffractometer	5268 measured reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)	2942 independent reflections
$T_{min} = 0.304$, $T_{max} = 0.556$	1946 reflections with $I > 2\sigma(I)$
(expected range = 0.281–0.515)	$R_{int} = 0.022$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$	172 parameters
$wR(F^2) = 0.093$	H-atom parameters constrained
$S = 0.88$	$\Delta\rho_{max} = 0.52$ e Å^{-3}
2942 reflections	$\Delta\rho_{min} = -0.45$ e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C1—H1⋯O2^i	0.93	2.50	3.157 (4)	128

Symmetry code: (i) $x - 2$, $y - 1$, z.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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supplementary materials

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

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Comment

The title compound, (I), (Fig. 1), was prepared as part of our ongoing studies (Harrison *et al.*, 2007) of the non-linear optical (NLO) properties (Uchida *et al.*, 1998) and crystal structures of chalcone derivatives. Compound (I) is centrosymmetric, thus its second harmonic generation (SHG) response is zero.

The geometrical parameters for (I) mostly fall within their expected ranges (Allen *et al.*, 1995). The C4—C3—Br1 angle of 126.5 (13)° is somewhat obtuse, perhaps due to steric repulsion between Br1 and H6 (H···Br = 2.72 Å), as also seen in related structures (Harrison *et al.*, 2007). This may help to explain why Br1 is displaced from the C1—C4/S1 mean plane by 0.046 (4) Å.

The dihedral angles between the enone (C5/C6/C7/O1) fragment and its adjacent thienyl (C1—C4/S1) and benzene (C8—C13) rings in (I) are 12.7 (2)° and 2.8 (2)°, respectively. The dihedral angle between the thienyl and benzene ring systems is 13.20 (18)° A possible weak intermolecular C—H···O interaction (Table 1) resulting in [001] chains of molecules may help to establish the crystal packing in (I).

Experimental

4-Nitrobenzaldehyde (1.51 g, 0.01 mol) in ethanol (20 ml) was mixed with 1-(3-bromo-2-thienyl)ethanone (2.05 ml, 0.01 mol) in 20 ml e thanol and the mixture was treated with 8 ml of 10% KOH solution at 283 K and stirred for 8 h. The precipitate obtained was filtered, washed with ethanol and dried. Pale yellow rods of (I) were grown from a 1:1 (*v/v*) solution of acetone and toluene by slow evaporation (m.p.: 452–54 K). Analysis for $C_{13}H_8BrNO_3S$: Found (calculated): C 46.11 (46.19); H 2.34 (2.38); N 4.09 (4.14); S 9.40% (9.48%).

Refinement

The hydrogen atoms were geometrically placed (C—H = 0.93 Å) and refined as riding with $U_{iso}(H) = 1.2U_{eq}$(carrier).
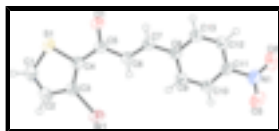
Figures

Fig. 1. View of the molecular structure of (I) showing 50% displacement ellipsoids (arbitrary spheres for the H atoms).

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Crystal data

$C_{13}H_8BrNO_3S$	$Z = 2$
$M_r = 338.17$	$F_{000} = 336$
Triclinic, $P\overline{1}$	$D_x = 1.727$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 4.8098$ (3) Å	Cell parameters from 2053 reflections
$b = 10.7863$ (8) Å	$\theta = 2.3–27.2°$
$c = 12.7709$ (9) Å	$\mu = 3.32$ mm^{-1}
$\alpha = 81.835$ (1)°	$T = 291$ (2) K
$\beta = 83.714$ (2)°	Rod, pale yellow
$\gamma = 85.316$ (2)°	$0.47 \times 0.20 \times 0.20$ mm
$V = 650.42$ (8) Å^3	

Data collection

Bruker SMART1000 CCD diffractometer	2942 independent reflections
Radiation source: fine-focus sealed tube	1946 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.022$
$T = 291(2)$ K	$\theta_{max} = 27.5°$
ω scans	$\theta_{min} = 2.3°$
Absorption correction: multi-scan (SADABS; Bruker, 1999)	$h = -5 \rightarrow 6$
$T_{min} = 0.304$, $T_{max} = 0.556$	$k = -13 \rightarrow 13$
5268 measured reflections	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.037$	H-atom parameters constrained
$wR(F^2) = 0.093$	$w = 1/[\sigma^2(F_o^2) + (0.0578P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.88$	$(\Delta/\sigma)_{max} = 0.001$
2942 reflections	$\Delta\rho_{max} = 0.52$ e Å^{-3}
172 parameters	$\Delta\rho_{min} = -0.45$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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 ($Å^2$)

	x	y	z	U_{iso}*/U_{eq}
C1	−0.0465 (7)	−0.0063 (3)	0.1850 (3)	0.0619 (9)
H1	−0.1649	−0.0656	0.1728	0.074*
C2	0.0182 (7)	0.0077 (3)	0.2825 (3)	0.0562 (8)
H2	−0.0480	−0.0410	0.3449	0.067*
C3	0.1991 (6)	0.1054 (2)	0.2781 (2)	0.0438 (6)
C4	0.2717 (6)	0.1638 (2)	0.1766 (2)	0.0405 (6)
C5	0.4353 (6)	0.2710 (3)	0.1287 (2)	0.0429 (6)
C6	0.6144 (5)	0.3285 (2)	0.1915 (2)	0.0436 (6)
H6	0.6465	0.2897	0.2594	0.052*
C7	0.7316 (5)	0.4352 (2)	0.1531 (2)	0.0418 (6)
H7	0.6924	0.4718	0.0854	0.050*
C8	0.9171 (5)	0.5005 (2)	0.2077 (2)	0.0379 (6)
C9	0.9742 (6)	0.4615 (3)	0.3122 (2)	0.0455 (7)
H9	0.8929	0.3910	0.3499	0.055*
C10	1.1483 (6)	0.5251 (3)	0.3609 (2)	0.0493 (7)
H10	1.1874	0.4977	0.4305	0.059*
C11	1.2645 (5)	0.6309 (3)	0.3043 (2)	0.0428 (6)
C12	1.2194 (6)	0.6710 (3)	0.2003 (2)	0.0469 (7)
H12	1.3056	0.7402	0.1627	0.056*
C13	1.0438 (6)	0.6065 (2)	0.1525 (2)	0.0450 (7)
H13	1.0087	0.6338	0.0825	0.054*
N1	1.4441 (5)	0.7013 (3)	0.3579 (2)	0.0567 (7)
O1	0.4159 (5)	0.3103 (2)	0.03438 (16)	0.0642 (6)
O2	1.5621 (5)	0.7873 (2)	0.3064 (2)	0.0839 (8)
O3	1.4633 (7)	0.6687 (3)	0.4522 (2)	0.0973 (9)
S1	0.10966 (17)	0.09492 (7)	0.08705 (6)	0.0547 (2)
Br1	0.31076 (8)	0.14643 (3)	0.40507 (2)	0.06575 (16)

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.072 (2)	0.0503 (18)	0.068 (2)	−0.0251 (16)	−0.0080 (18)	−0.0111 (15)

C2	0.064 (2)	0.0501 (17)	0.0533 (18)	−0.0197 (15)	−0.0024 (16)	0.0021 (14)
C3	0.0521 (17)	0.0389 (15)	0.0404 (14)	−0.0049 (12)	−0.0086 (13)	−0.0020 (11)
C4	0.0452 (15)	0.0408 (15)	0.0373 (13)	−0.0064 (12)	−0.0091 (12)	−0.0058 (11)
C5	0.0481 (16)	0.0449 (15)	0.0362 (14)	−0.0084 (12)	−0.0066 (12)	−0.0028 (11)
C6	0.0471 (16)	0.0489 (16)	0.0360 (13)	−0.0125 (13)	−0.0099 (12)	0.0000 (12)
C7	0.0435 (15)	0.0479 (15)	0.0349 (13)	−0.0091 (12)	−0.0067 (12)	−0.0032 (11)
C8	0.0335 (14)	0.0453 (15)	0.0352 (13)	−0.0051 (11)	−0.0016 (11)	−0.0061 (11)
C9	0.0494 (16)	0.0515 (16)	0.0357 (13)	−0.0182 (13)	−0.0029 (12)	0.0014 (12)
C10	0.0528 (17)	0.0669 (19)	0.0299 (13)	−0.0190 (15)	−0.0053 (12)	−0.0029 (12)
C11	0.0397 (15)	0.0487 (16)	0.0434 (15)	−0.0099 (12)	−0.0052 (12)	−0.0138 (12)
C12	0.0491 (17)	0.0448 (15)	0.0465 (15)	−0.0143 (13)	−0.0026 (13)	−0.0004 (13)
C13	0.0520 (17)	0.0470 (16)	0.0354 (13)	−0.0101 (13)	−0.0069 (13)	0.0023 (12)
N1	0.0549 (15)	0.0652 (17)	0.0562 (16)	−0.0158 (13)	−0.0076 (13)	−0.0210 (13)
O1	0.0828 (15)	0.0770 (15)	0.0369 (11)	−0.0382 (12)	−0.0176 (10)	0.0057 (10)
O2	0.0899 (18)	0.0788 (17)	0.0910 (19)	−0.0497 (15)	−0.0119 (15)	−0.0111 (14)
O3	0.123 (2)	0.123 (2)	0.0597 (16)	−0.0572 (18)	−0.0285 (16)	−0.0191 (16)
S1	0.0668 (5)	0.0564 (5)	0.0468 (4)	−0.0211 (4)	−0.0110 (4)	−0.0137 (3)
Br1	0.0878 (3)	0.0755 (3)	0.03571 (17)	−0.02357 (19)	−0.01421 (15)	0.00301 (14)

Geometric parameters (Å, °)

C1—C2	1.347 (5)	C7—H7	0.9300
C1—S1	1.692 (3)	C8—C9	1.392 (4)
C1—H1	0.9300	C8—C13	1.402 (3)
C2—C3	1.412 (4)	C9—C10	1.374 (4)
C2—H2	0.9300	C9—H9	0.9300
C3—C4	1.379 (4)	C10—C11	1.385 (4)
C3—Br1	1.884 (3)	C10—H10	0.9300
C4—C5	1.475 (4)	C11—C12	1.373 (4)
C4—S1	1.732 (3)	C11—N1	1.474 (3)
C5—O1	1.229 (3)	C12—C13	1.377 (4)
C5—C6	1.467 (4)	C12—H12	0.9300
C6—C7	1.329 (3)	C13—H13	0.9300
C6—H6	0.9300	N1—O2	1.202 (3)
C7—C8	1.469 (3)	N1—O3	1.218 (4)
C2—C1—S1	113.2 (2)	C9—C8—C7	122.8 (2)
C2—C1—H1	123.4	C13—C8—C7	119.0 (2)
S1—C1—H1	123.4	C10—C9—C8	121.2 (2)
C1—C2—C3	111.5 (3)	C10—C9—H9	119.4
C1—C2—H2	124.2	C8—C9—H9	119.4
C3—C2—H2	124.2	C9—C10—C11	118.7 (2)
C4—C3—C2	114.0 (3)	C9—C10—H10	120.7
C4—C3—Br1	126.50 (19)	C11—C10—H10	120.7
C2—C3—Br1	119.5 (2)	C12—C11—C10	122.0 (2)
C3—C4—C5	136.2 (2)	C12—C11—N1	119.5 (2)
C3—C4—S1	109.11 (19)	C10—C11—N1	118.5 (2)
C5—C4—S1	114.60 (19)	C11—C12—C13	118.7 (2)
O1—C5—C6	121.4 (2)	C11—C12—H12	120.7
O1—C5—C4	117.6 (2)	C13—C12—H12	120.7

C6—C5—C4	121.0 (2)	C12—C13—C8	121.2 (2)
C7—C6—C5	121.0 (2)	C12—C13—H13	119.4
C7—C6—H6	119.5	C8—C13—H13	119.4
C5—C6—H6	119.5	O2—N1—O3	123.8 (3)
C6—C7—C8	126.0 (2)	O2—N1—C11	118.5 (3)
C6—C7—H7	117.0	O3—N1—C11	117.7 (3)
C8—C7—H7	117.0	C1—S1—C4	92.20 (14)
C9—C8—C13	118.2 (2)		
S1—C1—C2—C3	0.8 (4)	C7—C8—C9—C10	−179.8 (3)
C1—C2—C3—C4	−0.6 (4)	C8—C9—C10—C11	0.9 (5)
C1—C2—C3—Br1	178.1 (2)	C9—C10—C11—C12	−2.5 (5)
C2—C3—C4—C5	176.2 (3)	C9—C10—C11—N1	178.1 (3)
Br1—C3—C4—C5	−2.5 (5)	C10—C11—C12—C13	2.7 (5)
C2—C3—C4—S1	0.2 (3)	N1—C11—C12—C13	−177.9 (3)
Br1—C3—C4—S1	−178.44 (16)	C11—C12—C13—C8	−1.3 (4)
C3—C4—C5—O1	−167.6 (3)	C9—C8—C13—C12	−0.2 (4)
S1—C4—C5—O1	8.2 (4)	C7—C8—C13—C12	180.0 (3)
C3—C4—C5—C6	12.4 (5)	C12—C11—N1—O2	−5.0 (4)
S1—C4—C5—C6	−171.8 (2)	C10—C11—N1—O2	174.4 (3)
O1—C5—C6—C7	9.2 (4)	C12—C11—N1—O3	174.8 (3)
C4—C5—C6—C7	−170.7 (3)	C10—C11—N1—O3	−5.8 (4)
C5—C6—C7—C8	−179.1 (2)	C2—C1—S1—C4	−0.6 (3)
C6—C7—C8—C9	−5.4 (5)	C3—C4—S1—C1	0.2 (2)
C6—C7—C8—C13	174.4 (3)	C5—C4—S1—C1	−176.7 (2)
C13—C8—C9—C10	0.4 (4)		

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···O2[i]	0.93	2.50	3.157 (4)	128

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Fig. 1