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(2*E*)-1-(5-Bromothiophen-2-yl)-3-(4-chlorophenyl)prop-2-en-1-one

H. D. Kavitha,[a] K. R. Roopashree,[b] Suresh B. Vepuri,[c] H. C. Devarajegowda[b]* and Venkatesh B. Devaru[d]

[a]Department of Physics, Govt. Science College, Hassan 573 201, Karnataka, India, [b]Department of Physics, Yuvaraja's College (Constituent College), University of Mysore, Mysore 570005, Karnataka, India, [c]Institute of Pharmacy, GITAM University, Visakhapatnam-45, Andhrapradesh, India, and [d]Department of Physics, P. G. Department of Physics, LVD College, Raichur 584103, Karnataka, India
Correspondence e-mail: devarajegowda@yahoo.com

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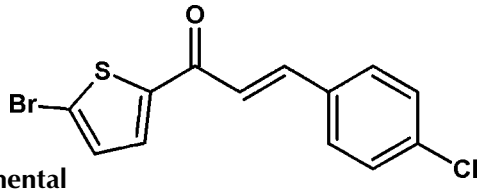
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.030; wR factor = 0.081; data-to-parameter ratio = 14.3.

In the title compound, $C_{13}H_8BrClOS$, the thiophene and phenyl rings are inclined by 40.69 (11)° to each other. The crystal structure is characterized by C—H$\cdots\pi$ interactions, which link the molecules into broad layers parallel to (100). Short Br$\cdots$Cl contacts [3.698 (1) Å] link these layers along [100].

Related literature

For general background to chalcones, see: Chun *et al.* (2001); Horng *et al.* (2003); Lopez *et al.* (2001); Mei *et al.* (2003). For related structures, see: Vepuri *et al.* (2012); Li & Su (1993).



Experimental

Crystal data

$C_{13}H_8BrClOS$
M_r = 327.61
Monoclinic, $P2_1/c$
a = 15.235 (3) Å
b = 13.959 (3) Å
c = 5.9153 (11) Å
β = 93.259 (3)°
V = 1255.9 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.63 mm^{-1}
T = 293 K
0.24 × 0.20 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2007)
T_{min} = 0.770, T_{max} = 1.000
14247 measured reflections
3032 independent reflections
2204 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.081
S = 1.05
3032 reflections
154 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

*Cg*2 is the centroid of the C5–C10 ring.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C10—H10$\cdots$*Cg*2^{i}	0.93	2.87	3.557 (3)	132
C16—H16$\cdots$*Cg*2^{ii}	0.93	2.96	3.480 (3)	117

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *SHELXL97*.

The authors thank Professor T. N. Guru Row, Soild State and Structural Chemistry Unit, Indian Institute of Science, Bangalore, for his constant support and for the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2505).

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supporting information

Acta Cryst. (2013). E**69**, o859 [doi:10.1107/S1600536813012063]

(2*E*)-1-(5-Bromothiophen-2-yl)-3-(4-chlorophenyl)prop-2-en-1-one

H. D. Kavitha, K. R. Roopashree, Suresh B. Vepuri, H. C. Devarajegowda and Venkatesh B. Devaru

S1. Comment

Chalcones are alpha beta unsaturated ketones, widely distributed in nature and are extensively studied for their biological activity (Chun *et al.*, 2001; Horng *et al.*, 2003; Lopez *et al.*, 2001; Mei *et al.*, 2003). We report here the crystal structure of a bromo derivative of hetero aryl chalcone which has shown aldose reductase inhibition in the virtual screening study conducted by us.

The titlecompound (2E)-1-(5-bromo-2-thienyl)-3-(4- chlorophenyl)prop-2-en-1-one, $C_{13}H_8Br$ Cl O S, presents a five-membered thiophene ring (S3\C14\···C17) and a phenyl ring (C5\C6\···C10) at 40.69 (11)° to each other (Fig 1). All intermolecular bond lengths and angles are within normal ranges (Vepuri *et al.*, 2012; Li & Su, 1993). The crystal structure is characterized by C—H···π interactions (C10—H10···Cg2; C16—H16···Cg2, Cg2 = C5->C10) (Table 1) which link molecules into broad 2D structures parallel to (100). There are in addition short intermolecular Br1···Cl2 contacts of 3.698 (1) Å, which link these structures along [100]. (Fig 2)

S2. Experimental

A mixture of 2-acetyl-5-BromoThiophene (0.01 mole) and 4-chlorobenzaldehyde (0.01 mole) were stirred in ethanol (30 ml) and then an aqueous solution of potassium hydroxide (40%,15 ml)was added to it. The mixture was kept over night at room temperature and then it was poured into crushed ice and acidified with dilute hydrochloric acid. The precipiteted chalcone was filtered and crystallized from ethanol.

S3. Refinement

All H atoms were positioned geometrically, with C—H = 0.93 Å for aromatic H and refined using a riding model with U_{iso}(H) = 1.2U_{eq}(C) for aromatic H.

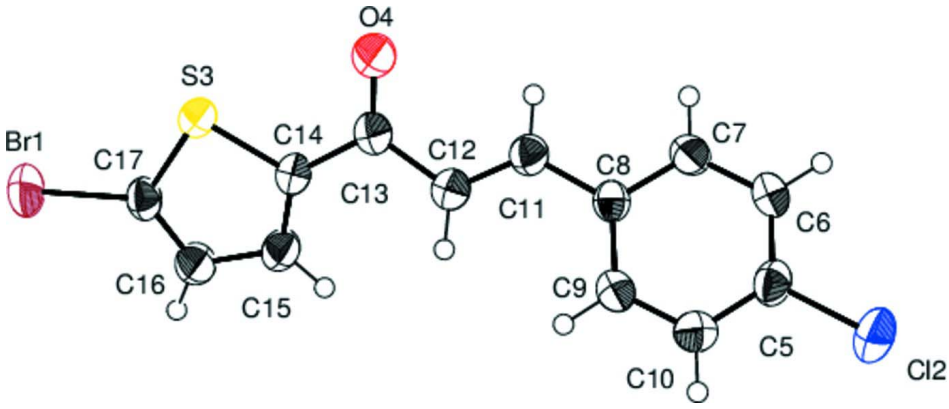

Figure 1

The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

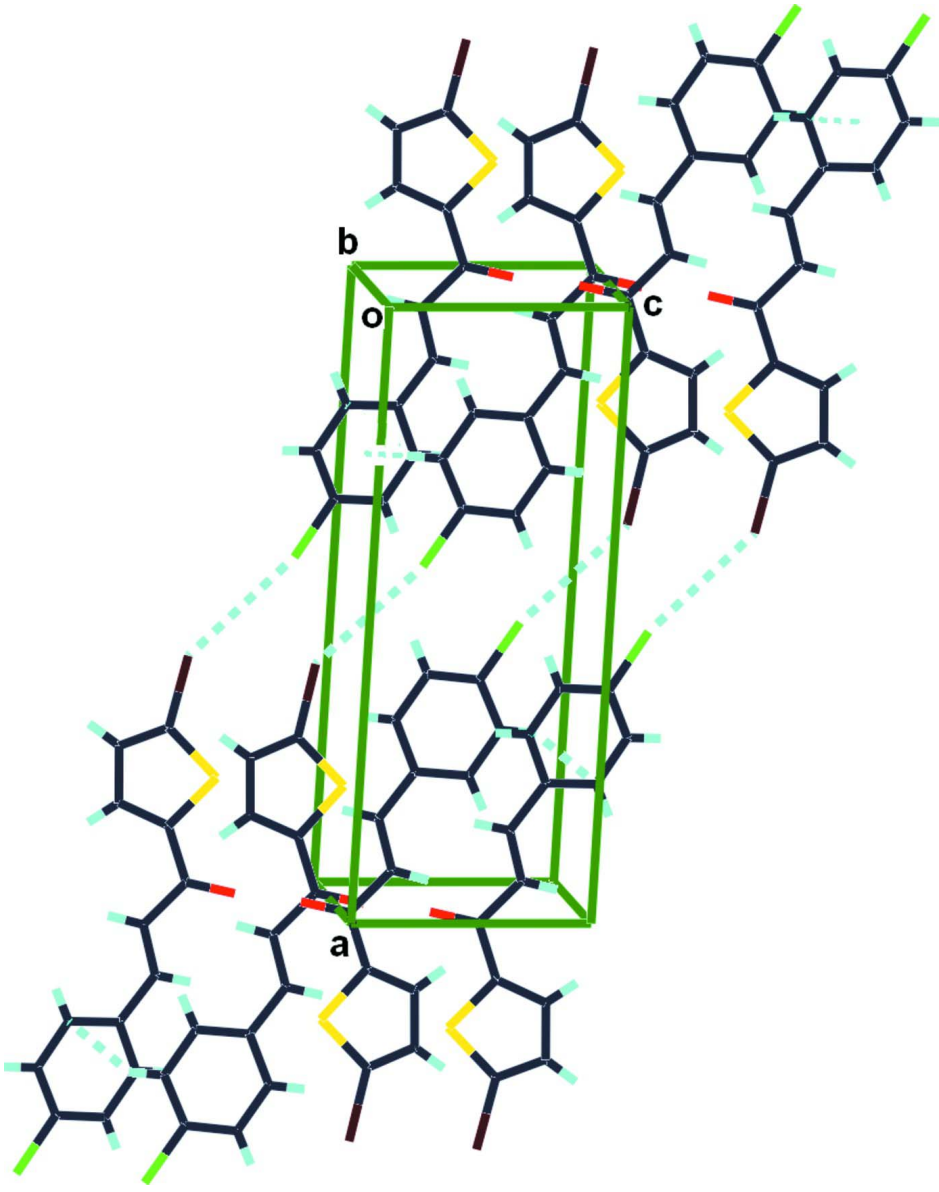

Figure 2

Crystal packing of the title compound. Dashed lines represent C-H···π and Br···Cl bonds.

(2*E*)-1-(5-Bromothiophen-2-yl)-3-(4-chlorophenyl)prop-2-en-1-one

Crystal data

$C_{13}H_8BrClOS$
$M_r = 327.61$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 15.235$ (3) Å
$b = 13.959$ (3) Å
$c = 5.9153$ (11) Å
$\beta = 93.259$ (3)°
$V = 1255.9$ (4) Å^3
$Z = 4$
$F(000) = 648$
$D_x = 1.733$ Mg m^{-3}
Melting point: 399 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2202 reflections
$\theta = 2.0$–25.0°
$\mu = 3.63$ mm^{-1}
$T = 293$ K
Plate, colourless
0.24 × 0.20 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2007)
T_{min} = 0.770, T_{max} = 1.000
14247 measured reflections
3032 independent reflections
2204 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 25.0°, θ_{min} = 2.0°
h = −18→18
k = 0→16
l = 0→7

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.081
S = 1.05
2202 reflections
154 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0473P)^2 + 0.2986P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
Br1	0.876810 (18)	0.34860 (2)	0.10591 (6)	0.06662 (15)
Cl2	0.03637 (5)	0.36156 (7)	0.68667 (14)	0.0719 (3)
S3	0.67904 (4)	0.34879 (5)	−0.04338 (10)	0.04481 (18)
O4	0.48985 (13)	0.37196 (16)	−0.1517 (3)	0.0642 (6)
C5	0.13007 (16)	0.37433 (18)	0.5358 (4)	0.0436 (6)
C6	0.12401 (15)	0.41523 (19)	0.3238 (4)	0.0466 (6)
H6	0.0705	0.4381	0.2626	0.056*
C7	0.19854 (15)	0.42171 (17)	0.2044 (4)	0.0421 (6)
H7	0.1943	0.4480	0.0598	0.051*
C8	0.28016 (15)	0.39015 (16)	0.2928 (4)	0.0369 (5)
C9	0.28356 (16)	0.35027 (16)	0.5098 (4)	0.0407 (6)
H9	0.3372	0.3292	0.5743	0.049*
C10	0.20961 (17)	0.34157 (16)	0.6295 (4)	0.0431 (6)
H10	0.2129	0.3138	0.7727	0.052*
C11	0.35582 (15)	0.39464 (17)	0.1545 (4)	0.0399 (5)
H11	0.3445	0.4112	0.0034	0.048*

C12	0.43893 (16)	0.37794 (19)	0.2184 (4)	0.0450 (6)
H12	0.4544	0.3669	0.3706	0.054*
C13	0.50768 (17)	0.37658 (18)	0.0522 (4)	0.0431 (6)
C14	0.59921 (15)	0.37793 (16)	0.1398 (4)	0.0371 (5)
C15	0.63572 (16)	0.40178 (19)	0.3477 (4)	0.0454 (6)
H15	0.6027	0.4194	0.4684	0.055*
C16	0.72770 (16)	0.39728 (19)	0.3626 (4)	0.0464 (6)
H16	0.7625	0.4121	0.4922	0.056*
C17	0.75950 (15)	0.36879 (17)	0.1654 (4)	0.0410 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.03415 (18)	0.0911 (3)	0.0755 (2)	0.00978 (13)	0.01087 (14)	0.00945 (16)
Cl2	0.0432 (4)	0.1058 (7)	0.0687 (5)	−0.0075 (4)	0.0196 (3)	0.0045 (4)
S3	0.0374 (4)	0.0601 (4)	0.0373 (3)	0.0041 (3)	0.0062 (3)	−0.0034 (3)
O4	0.0443 (11)	0.1042 (17)	0.0438 (11)	0.0027 (10)	−0.0004 (8)	−0.0067 (10)
C5	0.0348 (13)	0.0501 (14)	0.0466 (14)	−0.0074 (11)	0.0069 (11)	−0.0050 (12)
C6	0.0318 (13)	0.0592 (17)	0.0483 (15)	0.0033 (11)	−0.0024 (10)	0.0017 (12)
C7	0.0377 (13)	0.0474 (14)	0.0407 (13)	0.0003 (11)	−0.0025 (10)	0.0034 (11)
C8	0.0344 (12)	0.0350 (12)	0.0411 (13)	−0.0037 (10)	0.0003 (10)	−0.0021 (10)
C9	0.0351 (13)	0.0433 (14)	0.0431 (14)	0.0022 (10)	−0.0029 (11)	−0.0003 (10)
C10	0.0452 (14)	0.0432 (14)	0.0408 (14)	−0.0018 (11)	0.0013 (11)	0.0006 (10)
C11	0.0367 (13)	0.0429 (13)	0.0400 (13)	−0.0018 (10)	0.0020 (10)	−0.0024 (10)
C12	0.0369 (14)	0.0580 (15)	0.0402 (13)	0.0007 (11)	0.0039 (11)	0.0015 (11)
C13	0.0367 (13)	0.0484 (14)	0.0441 (15)	0.0010 (11)	0.0017 (11)	0.0003 (11)
C14	0.0320 (12)	0.0393 (13)	0.0406 (13)	0.0014 (10)	0.0077 (10)	−0.0008 (10)
C15	0.0424 (14)	0.0525 (16)	0.0420 (14)	0.0038 (11)	0.0077 (11)	−0.0078 (11)
C16	0.0407 (14)	0.0555 (16)	0.0430 (14)	−0.0036 (12)	0.0004 (11)	−0.0066 (12)
C17	0.0300 (12)	0.0426 (13)	0.0507 (14)	−0.0018 (10)	0.0042 (10)	0.0034 (11)

Geometric parameters (Å, º)

Br1—C17	1.863 (2)	C9—C10	1.370 (3)
Cl2—C5	1.735 (3)	C9—H9	0.9300
S3—C17	1.713 (3)	C10—H10	0.9300
S3—C14	1.723 (2)	C11—C12	1.321 (3)
O4—C13	1.223 (3)	C11—H11	0.9300
C5—C6	1.376 (4)	C12—C13	1.477 (3)
C5—C10	1.381 (4)	C12—H12	0.9300
C6—C7	1.374 (3)	C13—C14	1.460 (3)
C6—H6	0.9300	C14—C15	1.362 (3)
C7—C8	1.393 (3)	C15—C16	1.400 (3)
C7—H7	0.9300	C15—H15	0.9300
C8—C9	1.397 (3)	C16—C17	1.348 (3)
C8—C11	1.452 (3)	C16—H16	0.9300
C17—S3—C14	90.57 (12)	C12—C11—H11	116.2

C6—C5—C10	121.0 (2)	C8—C11—H11	116.2
C6—C5—Cl2	119.8 (2)	C11—C12—C13	121.1 (2)
C10—C5—Cl2	119.2 (2)	C11—C12—H12	119.5
C7—C6—C5	118.8 (2)	C13—C12—H12	119.5
C7—C6—H6	120.6	O4—C13—C14	120.3 (2)
C5—C6—H6	120.6	O4—C13—C12	122.1 (2)
C6—C7—C8	122.2 (2)	C14—C13—C12	117.6 (2)
C6—C7—H7	118.9	C15—C14—C13	131.0 (2)
C8—C7—H7	118.9	C15—C14—S3	111.04 (18)
C7—C8—C9	117.2 (2)	C13—C14—S3	117.92 (18)
C7—C8—C11	119.7 (2)	C14—C15—C16	113.7 (2)
C9—C8—C11	123.1 (2)	C14—C15—H15	123.2
C10—C9—C8	121.4 (2)	C16—C15—H15	123.2
C10—C9—H9	119.3	C17—C16—C15	111.5 (2)
C8—C9—H9	119.3	C17—C16—H16	124.3
C9—C10—C5	119.4 (2)	C15—C16—H16	124.3
C9—C10—H10	120.3	C16—C17—S3	113.23 (19)
C5—C10—H10	120.3	C16—C17—Br1	127.2 (2)
C12—C11—C8	127.6 (2)	S3—C17—Br1	119.61 (14)
C10—C5—C6—C7	1.0 (4)	C11—C12—C13—C14	167.3 (2)
Cl2—C5—C6—C7	−177.9 (2)	O4—C13—C14—C15	164.8 (3)
C5—C6—C7—C8	−1.4 (4)	C12—C13—C14—C15	−17.0 (4)
C6—C7—C8—C9	0.6 (4)	O4—C13—C14—S3	−12.8 (3)
C6—C7—C8—C11	177.1 (2)	C12—C13—C14—S3	165.31 (18)
C7—C8—C9—C10	0.7 (3)	C17—S3—C14—C15	0.3 (2)
C11—C8—C9—C10	−175.7 (2)	C17—S3—C14—C13	178.47 (19)
C8—C9—C10—C5	−1.1 (4)	C13—C14—C15—C16	−177.6 (2)
C6—C5—C10—C9	0.2 (4)	S3—C14—C15—C16	0.2 (3)
Cl2—C5—C10—C9	179.09 (18)	C14—C15—C16—C17	−0.8 (3)
C7—C8—C11—C12	171.9 (3)	C15—C16—C17—S3	1.0 (3)
C9—C8—C11—C12	−11.8 (4)	C15—C16—C17—Br1	−177.94 (19)
C8—C11—C12—C13	174.2 (2)	C14—S3—C17—C16	−0.8 (2)
C11—C12—C13—O4	−14.5 (4)	C14—S3—C17—Br1	178.26 (15)

Hydrogen-bond geometry (Å, º)

Cg2 is the centroid of the C5–C10 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10···*Cg*2^{i}	0.93	2.87	3.557 (3)	132
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