Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(2*E*)-1-(3-Bromo-2-thienyl)-3-(4-chlorophenyl)-prop-2-en-1-one, a twinned crystal structure

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.017 Å
R factor = 0.060
wR factor = 0.160
Data-to-parameter ratio = 14.1

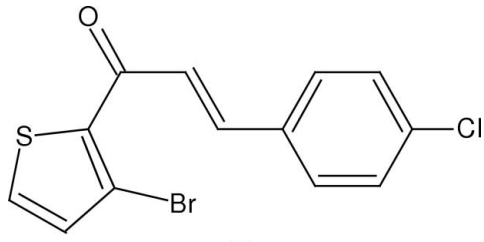
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 21 August 2006
Accepted 13 September 2006

Crystals of the title compound, $C_{13}H_{18}BrClOS$, appeared to be twinned by an interchange of the *a* and *c* axes. There are two almost identical molecules in the asymmetric unit which are essentially planar and do not show unusual geometric parameters.

Comment

Chalcones and their heterocyclic analogues show a number of biological activities (Opletalova & Sedivy, 1999). In addition, chalcones are a class of non-linear optical (NLO) materials (Fichou *et al.*, 1988; Goto *et al.*, 1991; Zhao *et al.*, 2000; Butcher *et al.*, 2006; Harrison *et al.*, 2006). Some similar prop-2-en-1-ones have been reported (Baxter *et al.*, 1990; Wang *et al.*, 2005; Patil *et al.*, 2006; Ng *et al.*, 2006). In continuation of our work on crystal structures of chalcones (Yathirajan *et al.*, 2006) and in view of their importance, the present paper reports the crystal structure of the title compound, (I).



(I)

A perspective view of (I) is shown in Fig. 1. Bond lengths and angles can be regarded as normal (Cambridge Structural Database, Version 5.27 of November 2005, updated August 2006; *MOGUL* Version 1.1; Allen, 2002). The two molecules in the asymmetric unit are almost identical. The dihedral angle between the least-squares planes through the non-H atoms of the two molecules is 52.91 (8)°. The central C=C double bond is *trans* configured. All non-H atoms lie almost in a common plane for each molecule (r.m.s. deviations of 0.114 and 0.049 Å for the two molecules in the asymmetric unit). A packing diagram (Fig. 2) reveals that the molecules show a preferred orientation, *i.e.* the C—Cl vectors are pointing either along *a* or along *c*. Thus, crystals of (I) could serve for NLO experiments, for which the absence of an inversion centre is a prerequisite.

Experimental

2-Acetyl-3-bromothiophene (10 g, 0.048 mol) in methanol (50 ml) was mixed with 4-chlorobenzaldehyde (6.7 g, 0.048 mol) and the mixture was treated with 10 ml of a 30% potassium hydroxide solu-

tion at 278 K. The reaction mixture was then brought to room temperature and stirred for 4 h. The precipitated solid was filtered off and washed with water, dried and recrystallized from ethyl acetate (yield 84%; m.p. 412–14 K). Analysis for $C_{13}H_8BrClOS$ found (calculated) (%): C 47.52 (47.66), H 2.38 (2.46).

Crystal data

$C_{13}H_8BrClOS$
M_r = 327.61
Monoclinic, *Pn*
a = 17.955 (2) Å
b = 3.9563 (3) Å
c = 17.973 (2) Å
β = 99.646 (9)°
V = 1258.7 (2) Å^3
Z = 4
D_x = 1.729 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 3.62 mm^{-1}
T = 173 (2) K
Plate, yellow
0.32 × 0.24 × 0.12 mm

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.390, T_{max} = 0.670
7621 measured reflections
4349 independent reflections
4201 reflections with $I > 2\sigma(I)$
R_{int} = 0.075
θ_{max} = 25.6°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.160
S = 1.07
4349 reflections
309 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1235P)^2 + 0.2142P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.90 e Å^{-3}
$\Delta\rho_{min}$ = −0.69 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0072 (18)
Absolute structure: Flack (1983), 2001 Friedel pairs
Flack parameter: −0.013 (16)

Data collection was carried out as usual because the frames did not show any signs of twinning or other warning signs. After problems were encountered during the structure solution, anisotropic refinement remained stalled at $R1$ = 0.16, although R_{int} and R_{sigma} looked promising. It was therefore assumed that the crystal was twinned. For a successful refinement the twin law (001/010/100) had to be applied, corresponding to exchange of the almost equal a and c axes. The ratio of the twin components refined to 0.347 (2)/0.653 (2). H atoms were found in a difference map, but positioned geometrically and allowed to ride on their parent C atoms at a distance of 0.95 Å and with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

SB thanks SJCE for financial assistance and BVA thanks Mangalore University for permission to carry out the research work.

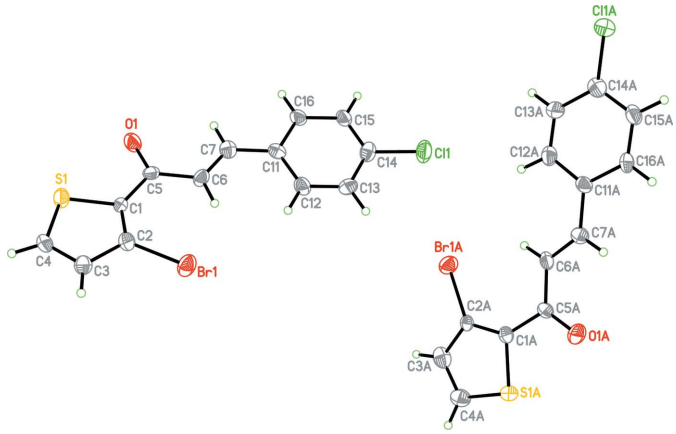


Figure 1
The asymmetric unit of the title compound with the atom numbering; displacement ellipsoids are drawn at the 50% probability level.

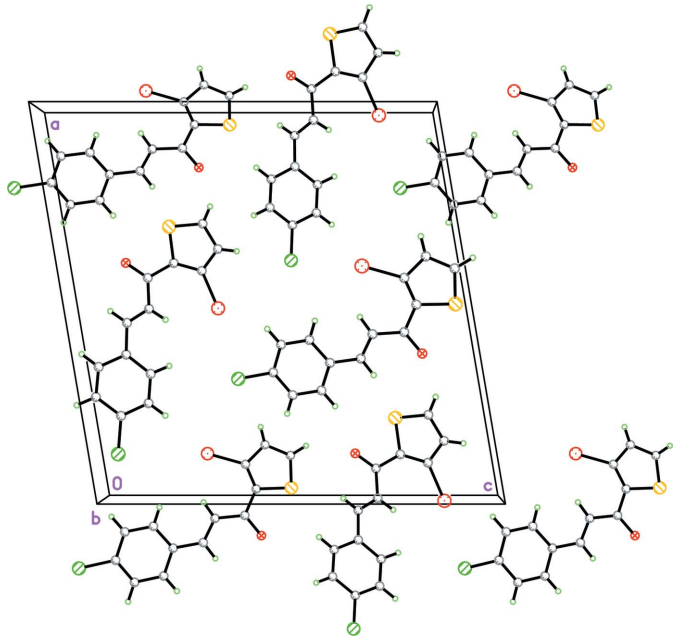


Figure 2
A packing diagram for the title compound, viewed down the b axis.

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