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(8*RS*,9*SR*)-Ethyl 4-(3-bromothien-2-yl)-6-(2-furyl)-2-oxocyclohex-3-ene-1-carboxylate

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Key indicators

Single-crystal X-ray study
T = 300 K
Mean σ(C–C) = 0.005 Å
R factor = 0.037
wR factor = 0.090
Data-to-parameter ratio = 16.3

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

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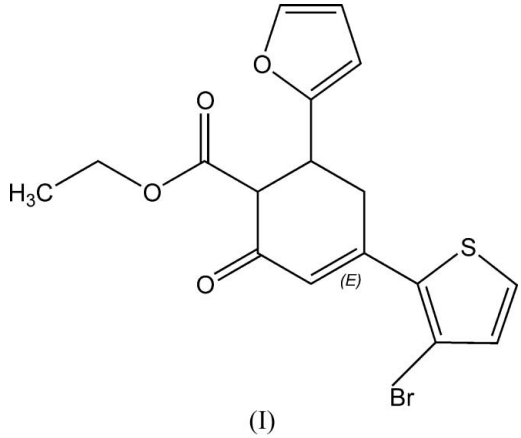
The title compound, $C_{17}H_{15}BrO_4S$, was synthesized from (2*E*)-1-(3-bromo-2-thienyl)-3-(2-furyl)prop-2-en-1-one and ethyl acetoacetate in an ethanol solution. Single crystals were obtained from an ethyl acetate/hexane mixture. The crystal packing is stabilized by van der Waals forces.

Comment

Chalcones and the corresponding heterocyclic analogs are valuable intermediates in organic synthesis (Dhar, 1981) and exhibit a multitude of biological activities (Dimmock *et al.*, 1999). From a chemical point of view, an important feature of chalcones and their heterocyclic analogs is their ability to act as activated unsaturated systems in conjugated addition reactions of carbanions in the presence of basic catalysts (House, 1972). This type of reaction may be exploited with the goal of obtaining highly functionalized cyclohexene derivatives (Tabba *et al.*, 1995), but is more commonly used for the preparation of 3,5-diaryl-6-carbethoxycyclohexenones *via* the Michael addition of ethyl acetoacetate. Cyclohexenones are efficient synthons in building spiro compounds (Padmavathi, Sharmila, Somashekara Reddy *et al.*, 2001) or intermediates in the synthesis of benzisoxazoles or carbazole derivatives (Padmavathi *et al.*, 1999, 2000; Padmavathi, Sharmila, Balaiah *et al.*, 2001). In view of the importance of these derivatives, a new compound, ethyl 3-(3-bromothien-2-yl)-6-(2-furyl)-2-oxocyclohex-3-ene-1-carboxylate, $C_{17}H_{15}BrO_4S$, (I), has been prepared and its crystal structure is reported here.



(I)

Compound (I) was prepared by the cyclocondensation of ethyl acetoacetate with a chalcone, leading to the generation of two chiral centers at C8 and C9 (Fig. 1). As the reaction is not stereoselective, both configurations of the chiral carbon atoms are expected to be found in (I), which would result in a

mixture of four diastereomers. In the title structure, a pair of 8*S*,9*R* and 8*R*,9*S* enantiomers is found. No attempt has been made to separate the diastereomers, and the crystal studied was grown from the mixture on recrystallization.

The bond lengths and angles of (I) are unexceptional. The cyclohexene ring adopts an envelope conformation, with C9 in the flap position. The dihedral angle between the thiophene and furan rings is 58.95 (14)°. One methyl group, C17, appears to exhibit slight disorder; however, refinement using a split model did not yield a satisfactory structural model. The crystal packing is stabilized by van der Waals forces.

Experimental

(2*E*)-1-(3-Bromo-2-thienyl)-3-(2-furyl)prop-2-en-1-one (1.42 g, 5 mmol) and ethyl acetoacetate (0.65 g, 5 mmol) were refluxed for 3 h in 15 ml of ethanol in the presence of 0.8 ml of 10% NaOH. The reaction mixture was cooled to 300 K. The product formed was filtered off and recrystallized from methanol. Crystals were grown from a (1:1) ethyl acetate–hexane solvent mixture (yield: 57%; m.p. 366–368 K). Analysis found (calculated) for $C_{17}H_{15}BrO_4S$ (%): C: 51.66 (51.64), H: 3.83 (3.81), S: 8.11 (8.09).

Crystal data

$C_{17}H_{15}BrO_4S$
M_r = 395.26
Monoclinic, $P2_1/c$
a = 16.5539 (14) Å
b = 11.1125 (8) Å
c = 9.1650 (5) Å
β = 100.098 (6)°
V = 1659.8 (2) Å^3
Z = 4
D_x = 1.582 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 2.62 mm^{-1}
T = 300 K
Irregular block, colourless
0.37 × 0.35 × 0.22 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
φ scans
Absorption correction: numerical (*HABITUS*; Herrendorf & Bärnighausen, 1997)
T_{min} = 0.600, T_{max} = 0.805
17471 measured reflections
3390 independent reflections
2655 reflections with $I > 2\sigma(I)$
R_{int} = 0.060
θ_{max} = 26.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.090
S = 1.17
3390 reflections
208 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.025P)^2 + 1.728P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}

H atoms were placed at calculated positions and refined using a riding model; U_{iso}(H) = 1.2U_{eq}(carrier atom), U_{iso}(H) = 1.5U_{eq}(methyl) and C—H = 0.93–0.98 Å.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *DIRAX/LSQ* (Duisenberg, 1992); data reduction: *EVALCCD* (Duisenberg *et al.*, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *maXus* (Mackay *et al.*, 1999).

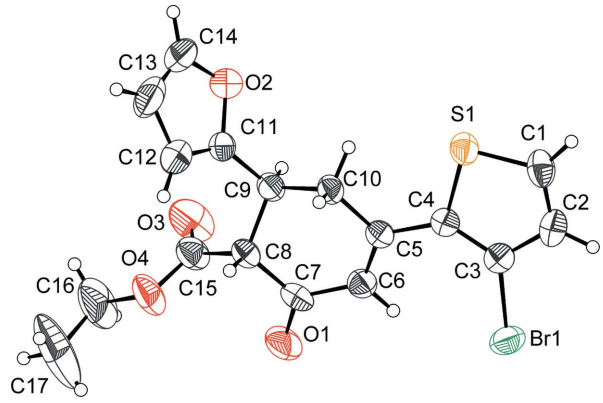


Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

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