

1-(3-Bromo-2-thienyl)-3-[4-(dimethylamino)-phenyl]prop-2-en-1-one

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Key indicators

Single-crystal X-ray study

$T = 273$ K

Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å

R factor = 0.033

wR factor = 0.086

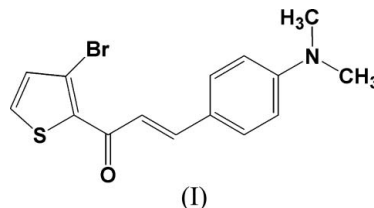
Data-to-parameter ratio = 17.4

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

The title compound, $\text{C}_{15}\text{H}_{14}\text{BrNOS}$, crystallizes with two molecules in the asymmetric unit. One of the two molecules forms dimers held together by weak $\text{C}-\text{H}\cdots\text{O}$ interactions. The twist angles between the thienyl and benzene rings are 2.70 (16) and 4.76 (18)°, smaller than usually observed in chalcone derivatives.

Comment

Among several organic compounds reported as having non-linear optical (NLO) properties, chalcone derivatives are notable materials for their excellent blue-light transmittance and good crystallizability. They provide a necessary configuration to show NLO properties with two planar rings connected through a conjugated double bond (Goto *et al.*, 1991; Uchida *et al.*, 1998; Tam *et al.*, 1989; Indira *et al.*, 2002). In a continuation of our work on chalcones (Butcher *et al.*, 2006, 2006*a,b*), the present paper reports the crystal structure of a newly synthesized chalcone, (I).



Unlike many other chalcone derivatives, which tend to be chiral due to the twist of the two planar rings, the title compound, (I), crystallizes in the centrosymmetric space group $P\bar{1}$, with two molecules in the asymmetric unit. The dihedral angle between the thienyl and phenyl planes in the two molecules are 2.70 (16) and 4.76 (18)°, smaller than the value usually observed in chalcone derivatives (Butcher *et al.*, 2006, 2006*a,b*). One of the two molecules in the asymmetric unit forms dimers which are linked by weak $\text{C}-\text{H}\cdots\text{O}$ interactions (Fig. 2 and Table 1). The metrical parameters of the thienyl and phenyl rings and the chalcone backbone in both molecules are within the normal ranges (Allen, 2002).

Experimental

2-Acetyl-3-bromothiophene (10 g, 0.048 mol) in methanol (50 ml) was mixed with 4-dimethylaminobenzaldehyde (7.16 g, 0.048 mol) and the mixture was treated with 10 ml of 30% potassium hydroxide solution at 278 K. The reaction mixture was then brought to room temperature and stirred for 4 h. The solid which precipitated was filtered and washed with water, dried and recrystallized from acetone (yield 70%; m.p. 371 K).

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Crystal data

C₁₅H₁₄BrNOS
M_r = 336.24
 Triclinic, *P*1̄
a = 9.6866 (3) Å
b = 12.6923 (4) Å
c = 12.8555 (5) Å
 α = 94.444 (1)°
 β = 101.673 (1)°
 γ = 107.322 (1)°

V = 1461.51 (9) Å³
Z = 4
D_x = 1.528 Mg m⁻³
 Mo *K*α radiation
 μ = 2.95 mm⁻¹
T = 273 (2) K
 Plate, colourless
 0.30 × 0.25 × 0.10 mm

Data collection

Bruker APEX-2 CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
T_{min} = 0.271, *T_{max}* = 0.590
 (expected range = 0.342–0.745)

13636 measured reflections
 6034 independent reflections
 4472 reflections with *I* > 2σ(*I*)
R_{int} = 0.020
 θ_{max} = 26.6°

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.033
wR (*F*²) = 0.086
S = 1.02
 6034 reflections
 347 parameters
 H-atom parameters constrained

w = 1/[σ²(*F_o*²) + (0.0395*P*)² + 0.3574*P*]
 where *P* = (*F_o*² + 2*F_c*²)/3
 (Δ/σ)_{max} = 0.001
 Δρ_{max} = 0.54 e Å⁻³
 Δρ_{min} = -0.43 e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C7A—H7AA...O1A ⁱ	0.93	2.52	3.404 (3)	159
C1B—H1BA...O1A ⁱⁱ	0.93	2.43	3.341 (4)	168

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

All H atoms were initially located in a difference Fourier map. The methyl H atoms were then constrained to an ideal geometry, with C—H = 0.98 Å and *U*_{iso}(H) = 1.5*U*_{eq}(C), but each group was allowed to rotate freely about its C—C bond. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.93–0.96 Å and *U*_{iso}(H) = 1.2*U*_{eq}(C).

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

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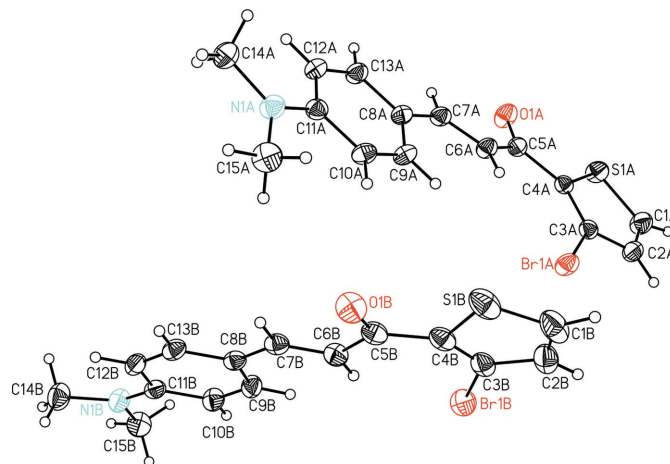


Figure 1

The asymmetric unit of (I) containing two molecules and showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

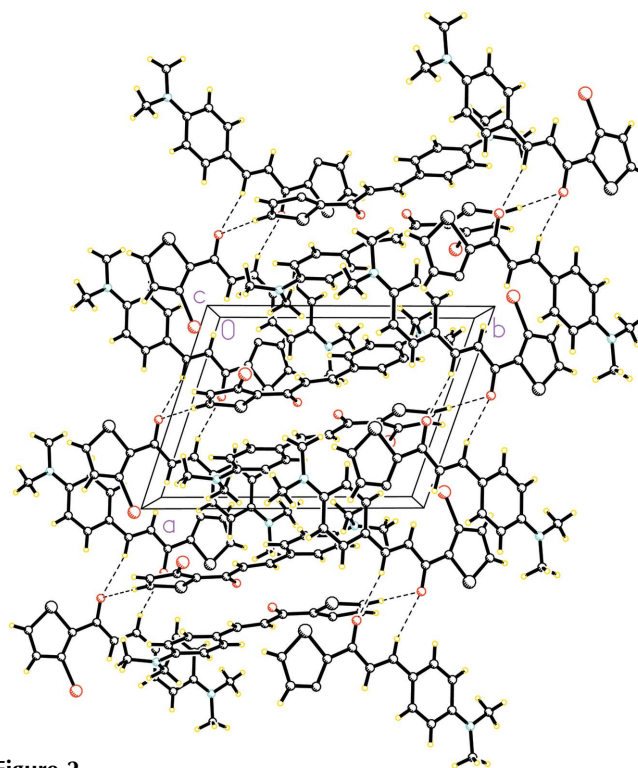


Figure 2

The molecular packing of (I), viewed down the *c* axis. Dashed lines indicate hydrogen bonds.

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