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Key indicators

Single-crystal X-ray study
 $T = 173$ K
Mean $\sigma(\text{C}-\text{C}) = 0.005$ Å
 R factor = 0.049
 wR factor = 0.127
Data-to-parameter ratio = 13.8

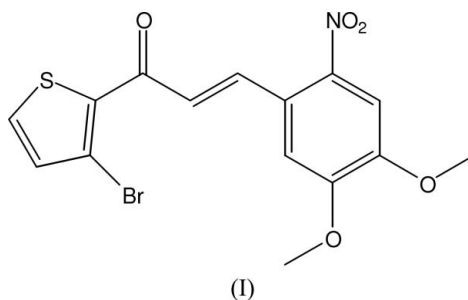
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

(2*E*)-1-(3-Bromo-2-thienyl)-3-(4,5-dimethoxy-2-nitrophenyl)prop-2-en-1-one

The title compound, $\text{C}_{15}\text{H}_{12}\text{BrNO}_5\text{S}$, is a chalcone derivative with potential nonlinear optical (NLO) properties. The molecule is essentially planar and does not show any unusual geometric parameters. The molecular structure and the crystal packing are characterized by $\text{C}-\text{H}\cdots\text{Br}$ and $\text{C}-\text{H}\cdots\text{O}$ contacts.

Comment

Among several organic compounds reported which have NLO properties, chalcone derivatives are recognized materials because of their excellent blue light transmittance and good crystallization ability. 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). Synthetic chalcones also have wide applications in the field of medicine. The crystal structures of 3-hydroxy-1,3-bis(2-thienyl)prop-2-en-1-one (Baxter *et al.*, 1990) and 1-(4-chlorophenyl)-3-(2-thienyl)prop-2-en-1-one (Ng *et al.*, 2006) have been reported. In continuation of our work on chalcones (Yathirajan *et al.*, 2006*a,b*), the present paper reports the crystal structure of the title compound, (I).



The molecular structure of (I) is shown in Fig. 1. Bond lengths and angles can be regarded as normal (Cambridge Structural Database; Version 5.27; November 2005 updated May 2006; Mogul Version 1.1; Allen, 2002). The central $\text{C}=\text{C}$ double bond is *trans* configured. All non-H atoms, except the two nitro O atoms of the molecule, lie almost in a common plane (r.m.s. deviation = 0.119 Å). The nitro group is rotated by 33.4 (2)° with respect to the benzene ring. The molecular structure and the crystal packing are characterized by $\text{C}-\text{H}\cdots\text{Br}$ and $\text{C}-\text{H}\cdots\text{O}$ contacts (Table 1).

Experimental

2-Acetyl-3-bromothiophene (10 g, 0.048 mol) in methanol (50 ml) was mixed with 3,4-dimethoxy-2-nitrobenzaldehyde (10.1 g,

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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 that precipitated was filtered off and washed with water, dried and recrystallized from acetone (yield 73%, m.p. 443–445 K). Analysis found (calculated) for $C_{15}H_{12}BrNO_5S$: C 45.19 (45.24), H 3.01 (3.04), N 3.47 (3.52)%.

Crystal data

$C_{15}H_{12}BrNO_5S$
 $M_r = 398.23$
 Monoclinic, $P2_1/c$
 $a = 8.9032$ (10) Å
 $b = 9.0892$ (7) Å
 $c = 19.307$ (2) Å
 $\beta = 95.174$ (9)°
 $V = 1556.0$ (3) Å³

$Z = 4$
 $D_x = 1.700$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 2.80$ mm⁻¹
 $T = 173$ (2) K
 Plate, brown
 $0.46 \times 0.43 \times 0.22$ mm

Data collection

Stoe IPDS-II two-circle diffractometer
 ω scans
 Absorption correction: multi-scan (MULABS; Spek, 2003; Blessing, 1995)

$T_{\min} = 0.319$, $T_{\max} = 0.578$
 (expected range = 0.298–0.540)
 8965 measured reflections
 2911 independent reflections
 2507 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.063$
 $\theta_{\text{max}} = 25.7^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.127$
 $S = 1.05$
 2911 reflections
 211 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0774P)^2 + 1.2577P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 1.22$ e Å⁻³
 $\Delta\rho_{\text{min}} = -1.07$ e Å⁻³
 Extinction correction: SHELXL97
 Extinction coefficient: 0.0082 (15)

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C6-H6\cdots Br1$	0.95	2.71	3.375 (4)	128
$C7-H7\cdots O5$	0.95	2.49	2.816 (4)	100
$C7-H7\cdots O12$	0.95	2.38	2.826 (4)	109
$C4-H4\cdots O18^i$	0.95	2.43	3.292 (5)	150
$C18-H18A\cdots O5^{ii}$	0.98	2.44	3.305 (5)	147
$C18-H18C\cdots O11^{iii}$	0.98	2.41	3.125 (4)	130

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

H atoms were found in a difference map, but placed geometrically and allowed to ride on their parent C atoms at distances of 0.95 and 0.98 Å for sp^2 and methyl groups, respectively, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$. The methyl groups were allowed to rotate but not to tip. The highest peak in the final

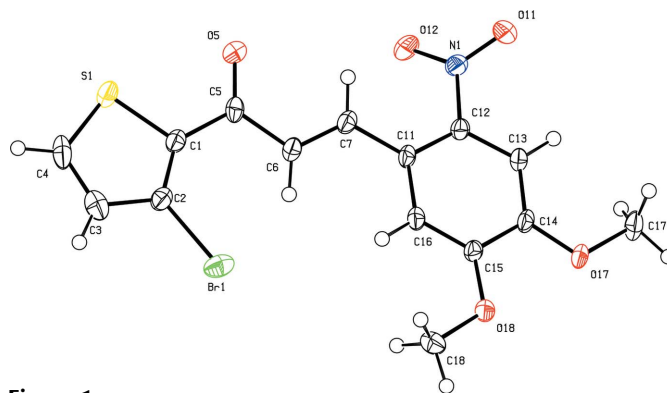


Figure 1

The molecular structure of (I) with the atom numbering; displacement ellipsoids are at the 50% probability level.

difference map is 0.76 Å from S1 and the deepest hole is 0.90 Å from atom Br1.

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: PLATON (Spek, 2003); software used to prepare material for publication: SHELXL97.

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