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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, least-squares-planes data and torsion angles have been deposited with the IUCr (Reference: FG1010). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Bednowitz, A. L. (1970). *Crystallographic Computing*, edited by F. R. Ahmed, S. R. Hall & C. P. Huber, pp. 58–62. Copenhagen: Munksgaard.
Brown, C. J. (1966). *Acta Cryst.* **21**, 153–158.
Carlisle, C. H. & Smith, C. H. (1971). *Acta Cryst.* B**27**, 1068–1069.
Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* A**24**, 321–324.
Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
Krigbaum, W. R., Chatani, Y. & Barber, P. G. (1970). *Acta Cryst.* B**26**, 97–102.
Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination*. Univ. of Cambridge, England.
Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

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3-(4-Bromophenyl)-1-(3-thienyl)-2-propen-1-one (BTC)

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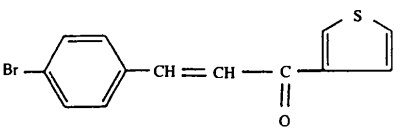
Abstract

The title chalcone derivative, $C_{13}H_9BrOS$, has a dihedral angle of 22.30° between the 4-bromobenzyl and the thienyl group planes. There is electron conjugation between the central —CH=CH—C(=O)— group and the benzyl and thienyl groups.

Comment

Chalcone derivatives are newly developed organic crystals with nonlinear optical properties (Kitaoka, Sasaki, Nakai & Goto, 1991). In an attempt to improve these properties, we have synthesized a series of substituted thiophene chalcone derivatives. Structural studies reveal that one of the products is the title compound, 3-(4-bromophenyl)-1-(3-thienyl)-2-propen-1-one, BTC.



BTC

In general, bond lengths in conjugated systems are intermediate between double- and single-bond lengths. For the title compound, BTC, the C(4)—C(7), C(9)—C(10), C(8)—C(9), C(7)—C(8) and O(1)—C(9) bond lengths are 1.47 (1), 1.49 (1), 1.483 (9), 1.29 (1) and 1.218 (7) Å, respectively. These bonds are similar to equivalent bonds found in 3-(4-chlorophenyl)-1-(3-thienyl)-2-propen-1-one (CTC) (He, Shi & Su, 1994). The C—Br distance is 1.893 (6) Å, longer than the C—Cl distance of 1.736 (4) Å in CTC. The dihedral angle between the planes of the 4-bromobenzyl group and the thienyl group is 22.30° (the equivalent dihedral angle in CTC is 21.93°). Both BTC and CTC crystallize in the same monoclinic system with space group $P2_1$. BTC exhibits nonlinear optical properties; this has been confirmed by a second harmonic generation efficiency measurement on a powder sample using the method of Kurtz & Perry (1968).

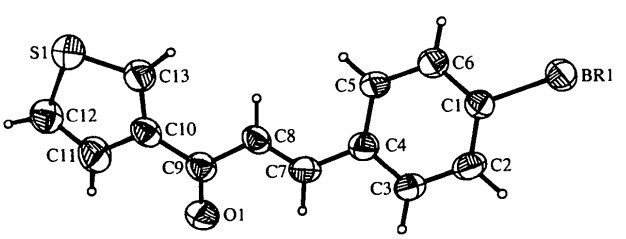


Fig. 1. The molecular structure of the title compound with the atomic numbering. The displacement ellipsoids are drawn at the 50% probability level.

Experimental

The title compound was prepared at room temperature by the condensation of 3-acetylthiophene and 4-bromobenzaldehyde in an alcoholic solution using sodium hydroxide as catalyst. A crystal was grown from alcoholic solution.

Crystal data

$C_{13}H_9BrOS$
$M_r = 293.18$
Monoclinic
$P2_1$
$a = 5.978\ (2)$ Å
$b = 4.945\ (2)$ Å
$c = 20.168\ (4)$ Å
$\beta = 95.80\ (3)°$
Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 14.8–16.8°$
$\mu = 3.57$ mm^{-1}
$T = 296$ K
Plate

$V = 593.2\ (6)\ Å^3$
$Z = 2$
$D_x = 1.64\ Mg\ m^{-3}$
$0.75 \times 0.75 \times 0.35$ mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: empirical *via* ψ scans (*TEXSAN*; Molecular Structure Corporation, 1985)
$T_{min} = 0.5252$, $T_{max} = 1.0000$
3062 measured reflections
2914 independent reflections
1529 observed reflections $[I > 3\sigma(I)]$
$R_{int} = 0.0213$
$\theta_{max} = 35°$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 7$
$l = -32 \rightarrow 32$
3 standard reflections
frequency: 60 min
intensity decay: 0.4%

Refinement

Refinement on F
$R = 0.046$
$wR = 0.052$
$S = 1.39$
1529 reflections
144 parameters
H-atom parameters not refined
$w = 1/\sigma^2(F_o)$
$(\Delta/\sigma)_{max} = 0.056$
$\Delta\rho_{max} = 0.42$ e $Å^{-3}$
$\Delta\rho_{min} = -0.72$ e $Å^{-3}$
Atomic scattering factors from Cromer & Waber (1974)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* ($Å^2$)

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Br(1)	0.12226 (9)	0.2857	0.55689 (3)	4.03 (2)
S(1)	0.5099 (3)	1.8778 (5)	0.9344 (1)	5.50 (9)
O(1)	1.0287 (7)	1.421 (1)	0.8002 (3)	5.6 (2)
C(1)	0.300 (1)	0.544 (1)	0.6074 (3)	3.2 (2)
C(2)	0.504 (1)	0.624 (2)	0.5858 (3)	4.0 (3)
C(3)	0.6349 (8)	0.811 (2)	0.6230 (3)	3.9 (2)
C(4)	0.567 (1)	0.913 (1)	0.6821 (3)	3.6 (2)
C(5)	0.362 (1)	0.827 (2)	0.7023 (3)	3.7 (3)
C(6)	0.231 (1)	0.639 (1)	0.6657 (3)	3.5 (2)
C(7)	0.712 (1)	1.106 (2)	0.7209 (3)	3.9 (3)
C(8)	0.665 (1)	1.249 (2)	0.7710 (3)	3.7 (3)
C(9)	0.830 (1)	1.428 (2)	0.8091 (3)	4.0 (3)
C(10)	0.749 (1)	1.613 (1)	0.8600 (3)	3.8 (2)
C(11)	0.892 (1)	1.796 (3)	0.8980 (3)	5.2 (3)
C(12)	0.787 (1)	1.953 (2)	0.9421 (3)	4.6 (3)
C(13)	0.535 (1)	1.639 (2)	0.8751 (3)	4.5 (3)

Table 2. *Selected geometric parameters* (Å, °)

Br(1)—C(1)	1.893 (6)	C(4)—C(7)	1.47 (1)
S(1)—C(13)	1.698 (8)	C(5)—C(6)	1.38 (1)
S(1)—C(12)	1.690 (7)	C(7)—C(8)	1.29 (1)
O(1)—C(9)	1.218 (7)	C(8)—C(9)	1.483 (9)
C(1)—C(6)	1.369 (8)	C(9)—C(10)	1.49 (1)
C(1)—C(2)	1.390 (9)	C(10)—C(13)	1.353 (9)
C(2)—C(3)	1.38 (1)	C(10)—C(11)	1.41 (1)
C(3)—C(4)	1.394 (9)	C(11)—C(12)	1.38 (1)
C(4)—C(5)	1.393 (8)		
C(13)—S(1)—C(12)	93.7 (4)	C(8)—C(7)—C(4)	127.7 (6)
C(6)—C(1)—C(2)	121.5 (6)	C(7)—C(8)—C(9)	123.2 (6)
C(6)—C(1)—Br(1)	119.4 (4)	O(1)—C(9)—C(8)	121.0 (7)
C(2)—C(1)—Br(1)	119.1 (5)	O(1)—C(9)—C(10)	120.3 (6)
C(3)—C(2)—C(1)	119.1 (6)	C(8)—C(9)—C(10)	118.6 (5)
C(4)—C(3)—C(2)	120.5 (5)	C(13)—C(10)—C(11)	110.5 (7)
C(3)—C(4)—C(5)	118.7 (6)	C(13)—C(10)—C(9)	126.3 (6)
C(3)—C(4)—C(7)	118.9 (5)	C(11)—C(10)—C(9)	123.2 (6)
C(5)—C(4)—C(7)	122.3 (6)	C(12)—C(11)—C(10)	115.1 (6)
C(6)—C(5)—C(4)	121.2 (6)	C(11)—C(12)—S(1)	108.6 (5)
C(5)—C(6)—C(1)	118.9 (5)	C(10)—C(13)—S(1)	112.1 (5)

A crystal of the title compound was mounted at a random orientation on a glass fibre. Data were collected with a scan width of $(0.55 + 0.35\ \tan\theta)°$, and corrected for Lorentz and polarization factors.

The structure was solved by direct methods using *MITHRIL* (Gilmore, 1983) and *DIRDIF* (Beurskens, 1984). H atoms were placed in geometrically calculated positions with C—H = 0.95 Å, but were not refined. The structure was refined on F using a full-matrix least-squares technique with anisotropic displacement parameters for the C, O, S and Br atoms. Anomalous-dispersion corrections were not applied.

Diffractometer software used: *CONTROL* (Molecular Structure Corporation, 1988). All calculations were performed on a MicroVAX II computer using the *TEXSAN* (Molecular Structure Corporation, 1985) program package. The view of the molecule was produced using *PLUTO* (Motherwell & Clegg, 1978).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and least-squares-planes data have been deposited with the IUCr (Reference: AB1178). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Beurskens, P. T. (1984). *DIRDIF. Direct Methods for Difference Structures – an Automatic Procedure for Phase Extension and Refinement of Difference Structure Factors*. Technical Report 1984/1. Crystallography Laboratory, Toernooiveld, 6525 ED Nijmegen, The Netherlands.

Cromer, D. T. & Waber, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Tables 2.2A, 2.3.1. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Gilmore, C. J. (1983). *MITHRIL. Computer Program for the Automatic Solution of Crystal Structures from X-ray Data*. Department of Chemistry, Univ. of Glasgow, Scotland.

He, Y., Shi, J. & Su, G. (1994). *Acta Cryst.* C**50**, 804–805.

Kitaoka, Y., Sasaki, T., Nakai, S. & Goto, Y. (1991). *Appl. Phys. Lett.* **59**, 19–21.

Kurtz, S. K. & Perry, T. T. (1968). *J. Appl. Phys.* **39**, 3798–3813.

Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Molecular Structure Corporation (1988). *CONTROL. An Automatic Package for Rigaku AFC Single-Crystal Diffractometers*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO. Program for Plotting Molecular and Crystal Structures*. Univ. of Cambridge, England.