Acta Cryst. (1994). C**50**, 804–805

3-(4-Chlorophenyl)-1-(3-thienyl)-2-propen-1-one

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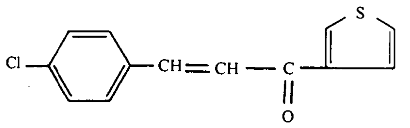
(*Received* 15 *April* 1993; *accepted* 7 *October* 1993)

Abstract

In 3-(4-chlorophenyl)-1-(3-thienyl)-2-propen-1-one (CTC) $C_{13}H_9ClOS$, the dihedral angle between the plane of the 4-chlorophenylpropenone group and the thienyl group is 18.45°. The molecule crystallizes in the non-centrosymmetric space group $P2_1$ and the crystals show large non-linear optical properties, as confirmed by powder second-harmonic generation (SHG) measurements.

Comment

3-(4-Methylphenyl)-1-(2-thienyl)-2-propen-1-one (MTC) (Kitaoka, Sasaki, Nakai & Goto, 1991) has a large second-harmonic generation (SHG) and also belongs to the monoclinic system with space group $P2_1$. We have synthesized a series of substituted thiophene chalcone derivatives. The title compound is one of them, which happens to crystallize in a non-centrosymmetric space group and is therefore likely to have non-linear optical properties. This has been confirmed by SHG efficiency measurements on a powder sample using the method of Kurtz & Perry (1968).



CTC

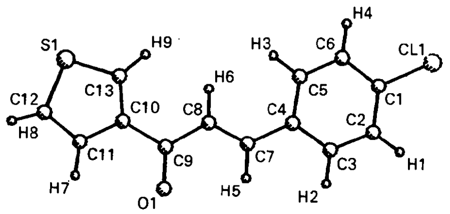


Fig. 1. Structure of the title molecule.

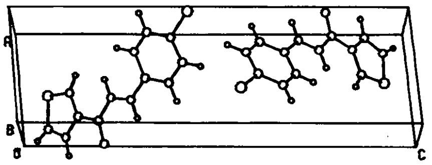


Fig. 2. Packing of the title molecules in the unit cell.

Experimental

The title compound was prepared by the condensation of NaOH on an alcoholic solution of 3-acetylthiopene and 4-chlorophenyl aldehyde at room temperature. A crystal was grown from an alcohol solution and mounted in a random orientation on a glass fibre.

Crystal data

$C_{13}H_9ClOS$
$M_r = 248.73$
Monoclinic
$P2_1$
$a = 5.942\ (1)$ Å
$b = 4.868\ (2)$ Å
$c = 20.104\ (5)$ Å
$\beta = 95.94\ (2)°$
$V = 578.4\ (3)$ Å^3
$Z = 2$

$D_x = 1.43$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 20 reflections
$\theta = 5–10°$
$\mu = 0.475$ mm^{-1}
$T = 296$ K
0.6 × 0.5 × 0.5 mm
Colourless

Data collection

Rigaku AFC-5*R* diffractometer
ω-2θ scans [width $(1.837 + 0.35\tan\theta)°$]
Absorption correction: empirical
$T_{\min} = 0.81$, $T_{\max} = 1.12$
2782 measured reflections
2650 independent reflections

2009 observed reflections [$I > 3\sigma(I)$]
$R_{\rm int} = 0.022$
$\theta_{\max} = 27°$
$h = 0 \rightarrow 8$
$k = -6 \rightarrow 6$
$l = -26 \rightarrow 26$
3 standard reflections
intensity variation: 1%

Refinement

Refinement on F
$R = 0.051$
$wR = 0.065$
$S = 1.64$
2009 reflections
144 parameters
H-atom parameters not refined

$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{\max} = 0.02$
$\Delta\rho_{\max} = 0.28$ e Å^{-3}
$\Delta\rho_{\min} = -0.30$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from Cromer & Waber (1974)

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

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

	x	y	z	$B_{\rm eq}$
Cl(1)	0.8790 (2)	0.3472	0.44555 (6)	4.17 (5)
S(1)	0.4937 (2)	−1.2349 (4)	0.06729 (7)	5.15 (6)
O(1)	−0.0224 (5)	−0.7809 (9)	0.2046 (2)	5.5 (2)
C(1)	0.7131 (7)	0.1082 (9)	0.3989 (2)	3.2 (2)
C(2)	0.5112 (7)	0.022 (1)	0.4221 (2)	3.5 (2)
C(3)	0.3794 (6)	−0.164 (1)	0.3836 (2)	3.5 (2)
C(4)	0.4436 (6)	−0.2723 (8)	0.3246 (2)	2.9 (2)

ISSN 0108-2701

C(5)	0.6506 (6)	−0.181 (1)	0.3039 (2)	3.3 (2)
C(6)	0.7820 (7)	0.005 (1)	0.3404 (2)	3.4 (2)
C(7)	0.2951 (7)	−0.461 (1)	0.2841 (2)	3.4 (2)
C(8)	0.3449 (7)	−0.608 (1)	0.2335 (2)	3.4 (2)
C(9)	0.1788 (7)	−0.787 (1)	0.1953 (2)	3.7 (2)
C(10)	0.2546 (7)	−0.971 (1)	0.1435 (2)	3.4 (2)
C(11)	0.1089 (7)	−1.151 (1)	0.1055 (2)	4.6 (2)
C(12)	0.2112 (8)	−1.309 (1)	0.0603 (2)	4.4 (2)
C(13)	0.4716 (7)	−0.997 (1)	0.1270 (2)	3.8 (2)

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

Cl(1)—C(1)	1.736 (4)	C(4)—C(7)	1.463 (6)
S(1)—C(13)	1.682 (5)	C(5)—C(6)	1.358 (6)
S(1)—C(12)	1.708 (5)	C(7)—C(8)	1.302 (6)
O(1)—C(9)	1.229 (5)	C(8)—C(9)	1.469 (6)
C(1)—C(6)	1.381 (6)	C(9)—C(10)	1.479 (6)
C(1)—C(2)	1.396 (6)	C(10)—C(13)	1.370 (6)
C(2)—C(3)	1.380 (6)	C(10)—C(11)	1.402 (6)
C(3)—C(4)	1.387 (6)	C(11)—C(12)	1.377 (7)
C(4)—C(5)	1.411 (5)		
C(13)—S(1)—C(12)	93.2 (2)	C(8)—C(7)—C(4)	127.5 (4)
C(6)—C(1)—C(2)	121.0 (4)	C(7)—C(8)—C(9)	122.5 (4)
C(6)—C(1)—Cl(1)	120.1 (3)	O(1)—C(9)—C(8)	121.3 (4)
C(2)—C(1)—Cl(1)	118.8 (3)	O(1)—C(9)—C(10)	119.5 (4)
C(3)—C(2)—C(1)	117.9 (4)	C(8)—C(9)—C(10)	119.2 (4)
C(2)—C(3)—C(4)	122.6 (4)	C(13)—C(10)—C(11)	110.7 (4)
C(3)—C(4)—C(5)	117.2 (4)	C(13)—C(10)—C(9)	126.1 (4)
C(3)—C(4)—C(7)	120.8 (4)	C(11)—C(10)—C(9)	123.2 (4)
C(5)—C(4)—C(7)	121.9 (4)	C(12)—C(11)—C(10)	114.7 (4)
C(6)—C(5)—C(4)	121.4 (4)	C(11)—C(12)—S(1)	108.9 (3)
C(5)—C(6)—C(1)	119.8 (4)	C(10)—C(13)—S(1)	112.4 (3)

Data collection was performed with *CONTROL* (Molecular Structure Corporation, 1988) diffractometer control software. Data were corrected for Lorentz and polarization factors. The structure was solved by direct methods using *MITHRIL* (Gilmore, 1983) and *DIRDIF* (Beurskens, 1984), the C, O, S and Cl atoms being located in an *E* map. H atoms were placed in geometrically calculated positions with C—H = 0.95 Å, but were not included in the refinement. The structure was refined by full-matrix least-squares techniques with anisotropic displacement parameters for C, O, S and Cl atoms. Anomalous dispersion-corrections were not applied. All calculations were performed on a MicroVAX II computer using the *TEXSAN* (Molecular Structure Corporation, 1985) program package. The views of the molecule and unit cell (Figs. 1 and 2, respectively) were produced using *PLUTO* (Motherwell & Clegg, 1978).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and bond distances involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71720 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HU1058]

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Acta Cryst. (1994). C**50**, 805–807

2-(*p*-Butoxyphenyl)-5-pentyl-1,3-dithiane, $C_{19}H_{30}OS_2$

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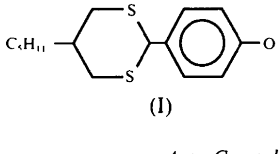
(*Received* 27 *April* 1993; *accepted* 19 *October* 1993)

Abstract

The molecules, which are *trans* isomers, are almost linearly extended and are stacked with their axes alternately oriented. The dithiane rings in the molecules are in chair conformations and are situated opposite each other.

Comment

2,5-Disubstituted 1,3-dithianes [R-($C_4H_6S_2$)(C_6H_4)-R'] have been reported as a new type of liquid-crystal compound (Haramoto & Kamogawa, 1983, 1985*a*,*b*; Haramoto, Nobe & Kamogawa, 1984; Haramoto, Akazawa & Kamogawa, 1984). The title compound (I) with $R = n$-C_5H_{11} and $R' = n$-OC_4H_9 exhibits monotropic behaviour and the nematic liquid-crystal phase appears in the temperature range 316–299 K.



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Acta Crystallographica Section C
ISSN 0108-2701 ©1994