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(2*E*)-1-(4-Chlorophenyl)-3-[4-(methylsulfanyl)-phenyl]prop-2-en-1-one

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.004 Å
R factor = 0.041
wR factor = 0.087
Data-to-parameter ratio = 17.1

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

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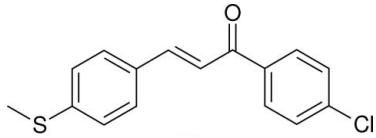
The geometrical parameters for the title compound, $C_{16}H_{13}ClOS$, are normal. The dihedral angle between the two benzene rings is 48.16 (5)°. The non-centrosymmetric crystal packing is consistent with the substantial non-zero second harmonic generation response.

Comment

The title compound, (I) (Fig. 1), was prepared as part of our ongoing studies (Harrison *et al.*, 2005; Harrison, Yathirajan, Sarojini *et al.*, 2006) of the non-linear optical (NLO) properties and crystal structures of chalcone derivatives. It is known that substitution at either benzene ring of the chalcone skeleton substantially affects optical response (Uchida *et al.*, 1998) and we are now exploring the role of the methylsulfanyl (H_3CS–) substituent (Harrison, Yathirajan, Mithun *et al.*, 2006) in this process.



(I)

Compound (I) displays a substantial second harmonic generation (SHG) response to red light of 7.4 times that of urea (Watson *et al.*, 1993). This is consistent with its polar space group. The geometrical parameters for (I) fall within their expected ranges (Allen *et al.*, 1987). The molecule of (I) is distinctly twisted about the C6—C7 and C9—C10 bonds (Table 1). The dihedral angle between the benzene ring best planes (C1–C6 and C10–C15) in (I) is 48.16 (5)°, which is similiar to the equivalent angle of 45.84 (4)° in 3-[4-(methylsulfanyl)phenyl]-1-(4-nitrophenyl)prop-2-en-1-one (Harrison, Yathirajan, Mithun *et al.*, 2006), but substantially smaller than the 68.15 (6)° seen in 2-bromo-1-(4-methylphenyl)-3-[4-(methylsulfanyl)phenyl]prop-2-en-1-one (Butcher *et al.*, 2006). The C16-methyl group in (I) is slightly displaced from the C10–C15 benzene ring mean plane [deviation = 0.169 (5) Å].

A *PLATON* (Spek, 2003) analysis of (I) indicated a possible intermolecular C—H···O interaction (Table 2) that might help to establish the crystal packing, which results in columns of molecules propagating in [001] with all the molecules aligned in the same sense with respect to the polar axis. Then, side-by-side [001] columns of molecules form pseudo-sheets in (010) (Fig. 2). This packing motif is very similar to that seen in 3-[4-(methylsulfanyl)phenyl]-1-(4-nitrophen-

yl)prop-2-en-1-one (Harrison, Yathirajan, Mithun *et al.*, 2006), although the overall symmetries and unit cells are quite different for these two phases.

Experimental

Aa aqueous solution of potassium hydroxide (5%, 5 ml) was added slowly with stirring to a mixture of 4-(methylsulfanyl)benzaldehyde (1.52 g, 0.01 mol) and 4-chloroacetophenone (1.54 g, 0.01 mol) in ethanol (15 ml). The resulting mixture was stirred at room temperature for 24 h. The precipitated solid was filtered off, washed with water, dried and plates of (I) were recrystallized from a (1:1 *v*/*v*) acetone–toluene mixture (yield 81%; m.p. 415–417 K). Analysis found (calculated) for $C_{16}H_{13}ClOS$ (%): C 66.41 (66.54), H 4.46 (4.54).

Crystal data

$C_{16}H_{13}ClOS$
M_r = 288.77
Monoclinic, *Cc*
a = 33.371 (2) Å
b = 6.9767 (5) Å
c = 5.8228 (3) Å
β = 90.376 (4)°
V = 1355.63 (14) Å^3
Z = 4
D_x = 1.415 Mg m^{-3}
Mo *K*α radiation
μ = 0.42 mm^{-1}
T = 120 (2) K
Plate, colourless
0.48 × 0.24 × 0.04 mm

Data collection

Nonius KappaCCD diffractometer
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)
T_{min} = 0.823, T_{max} = 0.983
8628 measured reflections
2964 independent reflections
2634 reflections with $I > 2\sigma(I)$
R_{int} = 0.047
θ_{max} = 27.6°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.087
S = 1.05
2964 reflections
173 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0314P)^2 + 0.6117P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Absolute structure: Flack (1983), 1399 Friedel pairs
Flack parameter: 0.07 (6)

Table 1
Selected torsion angles (°).

C5—C6—C7—O1	−21.6 (3)	C8—C9—C10—C11	−7.6 (4)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···O1^{i}	0.95	2.59	3.319 (3)	134

Symmetry code: (i) *x*, *y*, *z* + 1.

The H atoms were positioned geometrically (C—H = 0.95–0.98 Å) and refined as riding, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). The methyl group was allowed to rotate but not to tip to best fit the electron density.

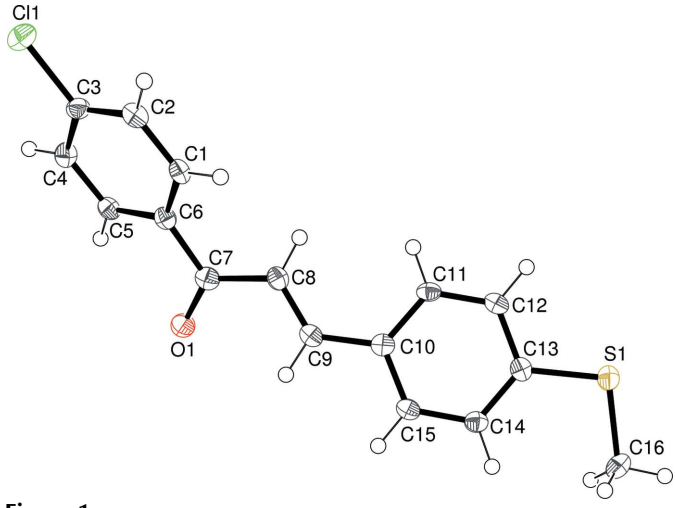


Figure 1
View of (I) showing 50% displacement ellipsoids (arbitrary spheres for the H atoms).

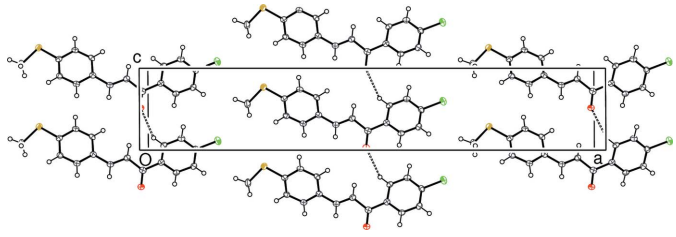


Figure 2
Detail of (I) showing side-by-side [001] columns forming an (010) pseudo-sheet. The C—H···O interactions are shown as dashed lines.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997), and *SORTAV* (Blessing, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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$c = 5.8228\ (3)$ Å
$\beta = 90.376\ (4)°$
$V = 1355.63\ (14)$ Å^3
$Z = 4$
$F(000) = 600$
$D_x = 1.415$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1626 reflections
$\theta = 2.9–27.5°$
$\mu = 0.42$ mm^{-1}
$T = 120$ K
Plate, colourless
0.48 × 0.24 × 0.04 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
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$T_{min} = 0.823$, $T_{max} = 0.983$
8628 measured reflections
2964 independent reflections
2634 reflections with $I > 2\sigma(I)$
$R_{int} = 0.047$
$\theta_{max} = 27.6°$, $\theta_{min} = 3.0°$
$h = -43\rightarrow42$
$k = -9\rightarrow9$
$l = -7\rightarrow7$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.087$
$S = 1.05$
2964 reflections
173 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0314P)^2 + 0.6117P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}
Absolute structure: Flack (1983), 1399 Friedel pairs
Absolute structure parameter: 0.07 (6)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.54021 (8)	0.8115 (3)	0.5478 (4)	0.0204 (5)
H1	0.5184	0.8519	0.6400	0.025*
C2	0.57921 (9)	0.8272 (4)	0.6312 (5)	0.0220 (6)
H2	0.5843	0.8802	0.7789	0.026*
C3	0.61055 (8)	0.7644 (4)	0.4957 (4)	0.0216 (5)
C4	0.60414 (8)	0.6857 (4)	0.2795 (4)	0.0221 (6)
H4	0.6259	0.6408	0.1900	0.026*
C5	0.56524 (8)	0.6744 (3)	0.1984 (4)	0.0203 (5)
H5	0.5604	0.6230	0.0498	0.024*
C6	0.53297 (9)	0.7367 (3)	0.3295 (4)	0.0192 (5)
C7	0.49173 (9)	0.7303 (3)	0.2283 (4)	0.0222 (6)
C8	0.45713 (9)	0.7309 (4)	0.3847 (5)	0.0214 (5)
H8	0.4607	0.6982	0.5419	0.026*
C9	0.42085 (8)	0.7773 (4)	0.3059 (4)	0.0194 (6)
H9	0.4195	0.8203	0.1512	0.023*
C10	0.38283 (8)	0.7699 (3)	0.4299 (4)	0.0194 (6)
C11	0.37873 (8)	0.6891 (3)	0.6506 (4)	0.0193 (5)
H11	0.4020	0.6474	0.7315	0.023*
C12	0.34175 (8)	0.6696 (4)	0.7509 (4)	0.0194 (6)
H12	0.3397	0.6145	0.8996	0.023*
C13	0.30687 (8)	0.7306 (3)	0.6350 (4)	0.0190 (5)
C14	0.31051 (8)	0.8129 (3)	0.4189 (4)	0.0207 (5)
H14	0.2873	0.8566	0.3391	0.025*
C15	0.34806 (8)	0.8313 (3)	0.3197 (4)	0.0194 (5)
H15	0.3500	0.8876	0.1717	0.023*
C16	0.22499 (9)	0.7720 (4)	0.5694 (5)	0.0328 (7)
H16A	0.1980	0.7445	0.6258	0.049*
H16B	0.2295	0.7021	0.4259	0.049*
H16C	0.2277	0.9099	0.5417	0.049*
O1	0.48761 (7)	0.7269 (3)	0.0189 (3)	0.0286 (5)
S1	0.26129 (2)	0.69787 (9)	0.77988 (9)	0.02479 (17)
Cl1	0.65933 (2)	0.78324 (11)	0.60236 (9)	0.03329 (19)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0240 (14)	0.0178 (12)	0.0196 (13)	−0.0004 (10)	0.0048 (10)	0.0017 (9)
C2	0.0303 (18)	0.0164 (13)	0.0194 (13)	−0.0007 (12)	0.0000 (11)	−0.0022 (11)
C3	0.0204 (14)	0.0204 (13)	0.0238 (13)	−0.0012 (10)	−0.0038 (10)	0.0040 (10)
C4	0.0218 (14)	0.0189 (13)	0.0256 (13)	0.0013 (10)	0.0036 (11)	−0.0013 (10)
C5	0.0254 (14)	0.0180 (12)	0.0175 (12)	−0.0017 (10)	0.0013 (10)	−0.0006 (9)
C6	0.0229 (14)	0.0130 (11)	0.0217 (13)	−0.0003 (10)	0.0012 (10)	0.0017 (10)
C7	0.0265 (15)	0.0167 (12)	0.0234 (14)	−0.0006 (11)	−0.0012 (11)	−0.0003 (10)
C8	0.0236 (15)	0.0193 (12)	0.0214 (13)	−0.0018 (11)	0.0021 (11)	0.0014 (10)
C9	0.0260 (16)	0.0144 (13)	0.0178 (13)	−0.0018 (10)	−0.0002 (11)	−0.0008 (9)
C10	0.0231 (15)	0.0149 (12)	0.0204 (13)	−0.0002 (10)	−0.0016 (11)	−0.0027 (10)
C11	0.0218 (14)	0.0152 (12)	0.0207 (13)	−0.0002 (10)	−0.0059 (10)	0.0018 (9)
C12	0.0274 (18)	0.0147 (12)	0.0161 (13)	−0.0020 (12)	−0.0024 (11)	−0.0001 (10)
C13	0.0233 (14)	0.0145 (11)	0.0192 (13)	−0.0007 (10)	0.0023 (10)	−0.0036 (9)
C14	0.0225 (14)	0.0196 (13)	0.0201 (12)	0.0021 (11)	−0.0016 (10)	0.0002 (10)
C15	0.0256 (15)	0.0147 (12)	0.0179 (12)	−0.0002 (10)	−0.0015 (10)	0.0000 (9)
C16	0.0185 (15)	0.0423 (16)	0.0375 (16)	0.0030 (13)	−0.0010 (12)	0.0062 (14)
O1	0.0255 (12)	0.0417 (12)	0.0185 (10)	−0.0004 (10)	0.0000 (8)	−0.0006 (8)
S1	0.0227 (4)	0.0266 (4)	0.0251 (3)	−0.0007 (3)	0.0030 (3)	0.0013 (3)
Cl1	0.0237 (4)	0.0407 (4)	0.0354 (4)	−0.0031 (3)	−0.0060 (3)	−0.0009 (3)

Geometric parameters (Å, º)

C1—C2	1.391 (4)	C9—H9	0.9500
C1—C6	1.394 (3)	C10—C15	1.390 (4)
C1—H1	0.9500	C10—C11	1.411 (3)
C2—C3	1.385 (4)	C11—C12	1.376 (4)
C2—H2	0.9500	C11—H11	0.9500
C3—C4	1.388 (4)	C12—C13	1.407 (4)
C3—Cl1	1.743 (3)	C12—H12	0.9500
C4—C5	1.381 (4)	C13—C14	1.389 (3)
C4—H4	0.9500	C13—S1	1.759 (3)
C5—C6	1.394 (4)	C14—C15	1.389 (4)
C5—H5	0.9500	C14—H14	0.9500
C6—C7	1.494 (4)	C15—H15	0.9500
C7—O1	1.227 (3)	C16—S1	1.794 (3)
C7—C8	1.475 (4)	C16—H16A	0.9800
C8—C9	1.332 (4)	C16—H16B	0.9800
C8—H8	0.9500	C16—H16C	0.9800
C9—C10	1.465 (4)		
C2—C1—C6	120.3 (2)	C10—C9—H9	116.2
C2—C1—H1	119.8	C15—C10—C11	117.2 (2)
C6—C1—H1	119.8	C15—C10—C9	119.0 (2)
C3—C2—C1	119.0 (2)	C11—C10—C9	123.5 (2)
C3—C2—H2	120.5	C12—C11—C10	121.3 (2)

C1—C2—H2	120.5	C12—C11—H11	119.4
C2—C3—C4	121.9 (3)	C10—C11—H11	119.4
C2—C3—Cl1	118.7 (2)	C11—C12—C13	120.5 (2)
C4—C3—Cl1	119.4 (2)	C11—C12—H12	119.7
C5—C4—C3	118.1 (2)	C13—C12—H12	119.7
C5—C4—H4	120.9	C14—C13—C12	118.8 (3)
C3—C4—H4	120.9	C14—C13—S1	124.7 (2)
C4—C5—C6	121.5 (2)	C12—C13—S1	116.47 (19)
C4—C5—H5	119.2	C15—C14—C13	119.9 (2)
C6—C5—H5	119.2	C15—C14—H14	120.0
C5—C6—C1	119.1 (2)	C13—C14—H14	120.0
C5—C6—C7	119.2 (2)	C14—C15—C10	122.2 (2)
C1—C6—C7	121.6 (2)	C14—C15—H15	118.9
O1—C7—C8	122.0 (3)	C10—C15—H15	118.9
O1—C7—C6	119.3 (2)	S1—C16—H16A	109.5
C8—C7—C6	118.6 (2)	S1—C16—H16B	109.5
C9—C8—C7	120.1 (2)	H16A—C16—H16B	109.5
C9—C8—H8	120.0	S1—C16—H16C	109.5
C7—C8—H8	120.0	H16A—C16—H16C	109.5
C8—C9—C10	127.6 (2)	H16B—C16—H16C	109.5
C8—C9—H9	116.2	C13—S1—C16	102.55 (13)

C6—C1—C2—C3	−1.1 (4)	C7—C8—C9—C10	174.3 (2)
C1—C2—C3—C4	−0.2 (4)	C8—C9—C10—C15	177.7 (3)
C1—C2—C3—Cl1	−179.6 (2)	C8—C9—C10—C11	−7.6 (4)
C2—C3—C4—C5	1.3 (4)	C15—C10—C11—C12	0.9 (3)
Cl1—C3—C4—C5	−179.33 (18)	C9—C10—C11—C12	−173.9 (2)
C3—C4—C5—C6	−1.1 (4)	C10—C11—C12—C13	−0.1 (4)
C4—C5—C6—C1	−0.2 (4)	C11—C12—C13—C14	−0.8 (4)
C4—C5—C6—C7	176.9 (2)	C11—C12—C13—S1	179.86 (18)
C2—C1—C6—C5	1.3 (3)	C12—C13—C14—C15	0.9 (3)
C2—C1—C6—C7	−175.7 (2)	S1—C13—C14—C15	−179.79 (19)
C5—C6—C7—O1	−21.6 (3)	C13—C14—C15—C10	−0.1 (4)
C1—C6—C7—O1	155.4 (2)	C11—C10—C15—C14	−0.8 (4)
C5—C6—C7—C8	159.2 (2)	C9—C10—C15—C14	174.3 (2)
C1—C6—C7—C8	−23.8 (3)	C14—C13—S1—C16	5.5 (2)
O1—C7—C8—C9	−17.4 (4)	C12—C13—S1—C16	−175.16 (19)
C6—C7—C8—C9	161.8 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···O1^{i}	0.95	2.59	3.319 (3)	134

Symmetry code: (i) *x*, *y*, *z*+1.