

1-(4-Chlorophenyl)-3-(4-methoxyphenyl)-
prop-2-en-1-oneWilliam T. A. Harrison,^{a*}
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Key indicators

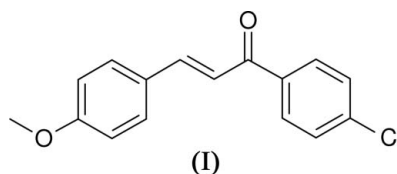
Single-crystal X-ray study
 $T = 120$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.029
 wR factor = 0.071
Data-to-parameter ratio = 16.3For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The geometrical parameters for the title compound, $\text{C}_{16}\text{H}_{13}\text{ClO}_2$, are normal. Packing in a non-centrosymmetric space group, which is consistent with the non-zero second harmonic generation response, may be influenced by a $\text{C}-\text{H} \cdots \text{O}$ interaction.

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Comment

The title compound, (I) (Fig. 1), was prepared as part of our ongoing studies (Indira *et al.*, 2002; Harrison *et al.*, 2005, 2006) of the non-linear optical (NLO) properties of chalcone derivatives (Uchida *et al.*, 1998). The non-centrosymmetric, polar crystal structure of (I) is consistent with its significant second harmonic generation (SHG) response of 0.8 times that of urea (Watson *et al.*, 1993).



The geometrical parameters for (I) are normal (Allen *et al.*, 1987) and consistent with those of other recently reported chalcone derivatives (Rosli *et al.*, 2006; Patil *et al.*, 2006). Compound (I) complements several closely related molecules with other 4-substituents X instead of Cl (see scheme), namely (II) with $X = \text{OH}$ (Moorthi *et al.*, 2005), (III) with $X = \text{CH}_3$ (Wang *et al.*, 2005), (IV) with $X = \text{H}$ (Rabinovich & Schmidt, 1970), and (V) with $X = \text{OCH}_3$ (Zheng *et al.*, 1992). The space groups for (I), (II), (III), (IV) and (V) are $Pna2_1$, $Pbca$, $P2_1/c$, $P2_1$ and $P2_12_12_1$, respectively. The distribution of space groups for this small family is thus consistent with the observation that chalcones are prone to crystallize as non-centrosymmetric structures (Uchida *et al.*, 1998).

The molecule of (I) is distinctly twisted about the C_6-C_7 and the C_7-C_8 bonds (Table 1), as was also seen for 2-bromo-1-chlorophenyl-3-(4-methoxyphenyl)-2-propen-1-one (Harrison *et al.*, 2006). The dihedral angle between the benzene ring mean planes (C_1-C_6 and $\text{C}_{10}-\text{C}_{15}$) in (I) is $21.82(6)^\circ$. $\text{Cl}1$, C_7 and $\text{O}1$ deviate from the former mean plane by 0.031 (3), 0.022 (3) and 0.346 (3) Å, respectively. The deviations of C_9 , $\text{O}2$ and C_{16} from the latter plane are 0.087 (3), 0.038 (3) and 0.049 (3) Å, respectively.

A *PLATON* (Spek, 2003) analysis of (I) indicated a possible intramolecular $\text{C}_9-\text{H}_9 \cdots \text{O}1$ interaction (Table 2) that might help to maintain the molecular conformation. A similar interaction was proposed for 3-(4-bromophenyl)-1-(4-nitrophenyl)-prop-2-en-1-one (Rosli *et al.*, 2006).

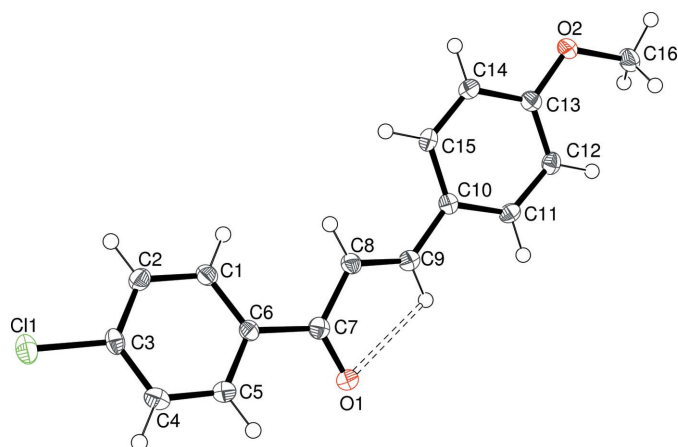


Figure 1
View of (I), showing 50% displacement ellipsoids (arbitrary spheres for the H atoms). The possible intramolecular C—H...O interaction is indicated by a dashed line.

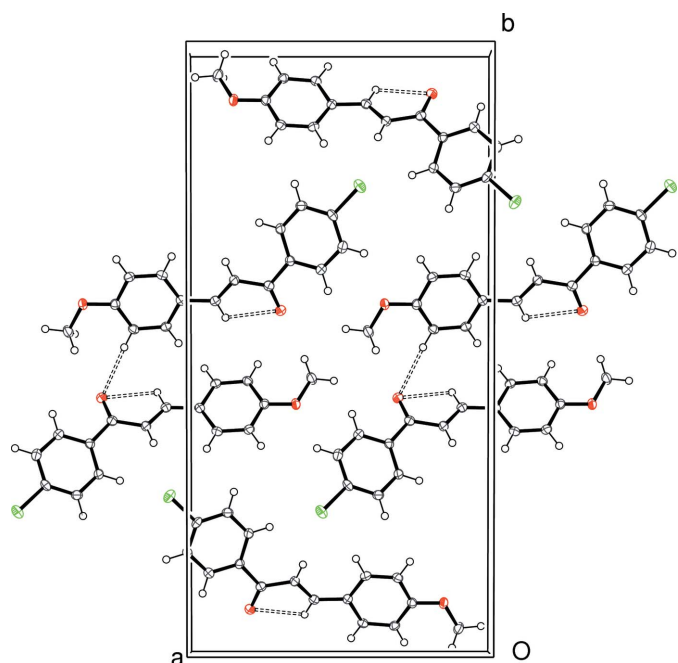


Figure 2
The packing for (I), viewed down [001], with C—H...O interactions shown as dashed lines.

An intermolecular C—H...O hydrogen bond (Fig. 2) appears to help to assemble the molecules of (I) into helical stacks about the 2_1 screw axis, propagating in the polar [001] direction.

Experimental

4-Chloroacetophenone in ethanol (1.54 g, 0.01 mol) (25 ml) was mixed with 4-methoxybenzaldehyde (1.36 g, 0.01 mol) in ethanol (25 ml) and the mixture was treated with an aqueous solution of potassium hydroxide (20 ml, 5%). This mixture was stirred well and left to stand for 24 hr. The resulting crude solid mass was collected by filtration and recrystallized from ethanol, yielding clear blocks of (I).

Yield: 90%, m.p.: 380 K, analysis found (calc.) for $C_{16}H_{13}ClO_2$, C 70.5 (70.4%); H 4.72 (4.76%).

Crystal data

$C_{16}H_{13}ClO_2$
 $M_r = 272.71$
Orthorhombic, $Pna2_1$
 $a = 12.8179$ (4) Å
 $b = 25.5550$ (6) Å
 $c = 3.9175$ (1) Å
 $V = 1283.22$ (6) Å³
 $Z = 4$
 $D_x = 1.412$ Mg m⁻³

Mo $K\alpha$ radiation
Cell parameters from 1708 reflections
 $\theta = 2.9$ – 27.5°
 $\mu = 0.29$ mm⁻¹
 $T = 120$ (2) K
Slab, colourless
 $0.50 \times 0.40 \times 0.20$ mm

Data collection

Nonius KappaCCD diffractometer
 ω and φ scans
Absorption correction: multi-scan (SADABS; Bruker, 2003)
 $T_{min} = 0.868$, $T_{max} = 0.944$
8514 measured reflections
2815 independent reflections

2569 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.028$
 $\theta_{max} = 27.5^\circ$
 $h = -9 \rightarrow 16$
 $k = -33 \rightarrow 32$
 $l = -5 \rightarrow 5$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.029$
 $wR(F^2) = 0.071$
 $S = 1.04$
2815 reflections
173 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0355P)^2 + 0.2552P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 0.23$ e Å⁻³
 $\Delta\rho_{min} = -0.22$ e Å⁻³
Absolute structure: Flack (1983),
1131 Friedel pairs
Flack parameter: 0.02 (6)

Table 1

Selected geometric parameters (Å, °).

C6—C7	1.496 (2)	C8—C9	1.342 (2)
C7—C8	1.479 (2)	C9—C10	1.461 (2)
C5—C6—C7—O1	16.7 (2)	O1—C7—C8—C9	6.4 (2)

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C9—H9...O1	0.95	2.46	2.8065 (19)	102
C12—H12...O1 ⁱ	0.95	2.54	3.4828 (18)	175

Symmetry code: (i) $-x + 1, -y, z - \frac{1}{2}$.

H atoms were positioned geometrically (C—H = 0.95–0.98 Å) and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(\text{carrier})$ or $1.5U_{eq}(\text{methyl carrier})$. The methyl group was rotated to fit the electron density.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor 1997); data reduction: *HKL DENZO* (Otwinowski & Minor 1997), *SCALEPACK* 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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References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–S19.
- Blessing, R. H. (1995). *Acta Cryst. A* **51**, 33–38.
- Bruker (2003). *SADABS*, Bruker AXS Inc., Madison, Wisconsin, USA.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565–565.
- Flack, H. D. (1983). *Acta Cryst. A* **39**, 876–881.
- Harrison, W. T. A., Yathirajan, H. S., Sarojini, B. K., Narayana, B. & Anilkumar, H. G. (2005). *Acta Cryst. C* **61**, o728–o730.
- Harrison, W. T. A., Yathirajan, H. S., Sarojini, B. K., Narayana, B. & Vijaya Raj, K. K. (2006). *Acta Cryst. E* **62**, o1578–o1579.
- Indira, J., Karat, P. P. & Sarojini, B. K. (2002). *J. Cryst. Growth*, **242**, 209–214.
- Moorthi, S. S., Chinnakali, K., Nanjundan, S., Radhika, R., Fun, H.-K. & Yu, X.-L. (2005). *Acta Cryst. E* **61**, o480–o482.
- Nonius (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
- Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter, Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
- Patil, P. S., Ng, S.-L., Razak, I. A., Fun, H.-K. & Dharmaparakash, S. M. (2006). *Acta Cryst. E* **62**, o1465–o1465.
- Rabinovich, D. & Schmidt, G. M. J. (1970). *J. Chem. Soc. B*, pp. 6–9.
- Rosli, M. M., Patil, P. S., Fun, H.-K., Razak, I. A. & Dharmaparakash, S. M. (2006). *Acta Cryst. E* **62**, o1466–o1468.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Uchida, T., Kozawa, K., Sakai, T., Aoki, M., Yoguchi, H., Abduryim, A. & Watanabe, Y. (1998). *Mol. Cryst. Liq. Cryst.* **315**, 135–140.
- Wang, L., Lu, C.-R., Zhang, Y. & Zhang, D.-C. (2005). *Jiegou Huaxue (Chin. J. Struct. Chem.)*, **24**, 191–195. (In Chinese.)
- Watson, G. J. R., Turner, A. B. & Allen, S. (1993). *Organic Materials for Non-linear Optics III*, edited by G. J. Ashwell & D. Bloor. RSC Special Publication No. 137, pp. 112–117.
- Zheng, J., Zhang, D., Sheng, P., Wang, H. & Yao, X. (1992). *Yingyong Huaxue (Chin. J. Appl. Chem.)*, **9**, 66–69. (In Chinese.)