organic papers

1-(4-Chlorophenyl)-3-(2,4,5-trimethoxyphenyl)-prop-2-en-1-one

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The title molecule, $C_{18}H_{17}ClO_4$, is slightly distorted from planarity. The dihedral angle between the two benzene rings is 11.6 (2)°. The crystal packing is stabilized by C—H···π interactions and van der Waals forces.

Comment

The synthesis and study of molecular compounds with nonlinear optical (NLO) properties has attracted much attention, because such materials hold promise for applications in optoelectronic and photonic devices (Bosshard *et al.*, 1995; Nalwa & Miyata, 1997). In order to create efficient quadratic (second-order) NLO materials, both the molecular and bulk properties must be optimized. The majority of promising compounds constitute dipolar donor–π-acceptor (D–π-A) molecules and these must be arranged non-centro-symmetrically in order to afford macroscopic structures capable of showing bulk quadratic NLO effects, such as frequency doubling (second-harmonic generation, SHG).

Within the diverse range of existing NLO compounds, chalcone derivatives are particularly attractive for their excellent blue light transmittance, good crystal stability, large nonlinear optical coefficients and relatively short cutoff wavelengths of transmittance (Goto *et al.*, 1991; Harrison *et al.*, 2005; Harrison, Yathirajan, Sarojini, Narayana & Vijaya Raj, 2006; Harrison, Yathirajan, Sarojini, Narayana & Indira, 2006; Patil *et al.*, 2006; Uchida *et al.*, 1998; Zhang *et al.*, 1990), much better than that observed in inorganic crystals. We have synthesized a new trimethoxy-substituted chalcone and have found it to possesses SHG efficiency. Its quantitative estimation is yet to be performed. In view of the importance of the title compound, (I), the crystal structure is reported here.

(I)
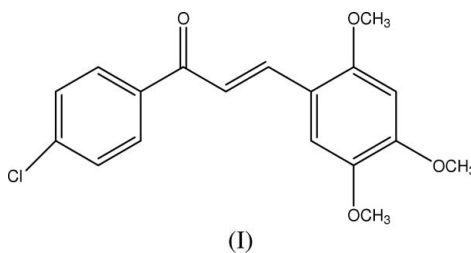
Bond lengths and angles in (I) have normal values (Allen *et al.*, 1987) and are comparable to those reported for related structures (Ng *et al.*, 2006; Patil *et al.*, 2006). The enone group (O1/C7–C9) and the two benzene rings (C1–C6 and C10–C15) of the chalcone are each planar, with maximum deviations of 0.011 (2), 0.011 (2) and 0.010 (2) Å for atoms C7, C1 and C13, respectively. The two methoxy groups attached at C12 and C13

are almost coplanar with the attached ring [C18—O4—C12—C11 = −6.6 (4)° and C17—O3—C13—C14 = 3.4 (5)°], while the methoxy group at C15 is twisted away from the C10–C15 benzene ring, with a C16—O2—C15—C14 torsion angle of −13.0 (5)°. The molecule is slightly twisted about the C9—C10 bond in (I), with a dihedral angle of 11.6 (2)° between the two benzene rings.

The crystal structure is stabilized by weak intermolecular C—H···π interactions (Table 1) involving the C1–C6 benzene ring (centroid $Cg1$) and van der Waals forces.

Experimental

4-Chloroacetophenone (0.01 mol) in ethanol (30 ml) was mixed with 2,4,5-trimethoxybenzaldehyde (0.01 mol) in ethanol (30 ml) and the mixture was treated with an aqueous solution of sodium hydroxide (5 ml, 20%). This mixture was stirred well and left for 12 h. The resulting crude solid mass was collected by filtration, dried and recrystallized from acetone.

Crystal data

$C_{18}H_{17}ClO_4$
$M_r = 332.77$
Orthorhombic, $P2_12_12_1$
$a = 7.0295$ (4) Å
$b = 10.5127$ (6) Å
$c = 22.2438$ (13) Å
$V = 1643.80$ (16) Å^3

$Z = 4$
$D_x = 1.345$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.25$ mm^{-1}
$T = 297$ (2) K
Needle, yellow
$0.53 \times 0.11 \times 0.08$ mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.825$, $T_{max} = 0.981$

15298 measured reflections
4783 independent reflections
2062 reflections with $I > 2\sigma(I)$
$R_{int} = 0.065$
$\theta_{max} = 30.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.139$
$S = 0.99$
4783 reflections
211 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0401P)^2 + 0.2277P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.13$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}
Absolute structure: Flack (1983), 2050 Friedel pairs
Flack parameter: 0.21 (11)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C18—H18B···$Cg1^i$	0.96	2.89	3.672 (4)	139

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

H atoms were placed in calculated positions, with C—H distances in the range 0.93–0.96 Å. The U_{iso}(H) values were set equal to $1.5U_{eq}$(C) for methyl H atoms and $1.2U_{eq}$(C) for the remaining H atoms. The value of the Flack parameter indicates possible partial inversion twinning.
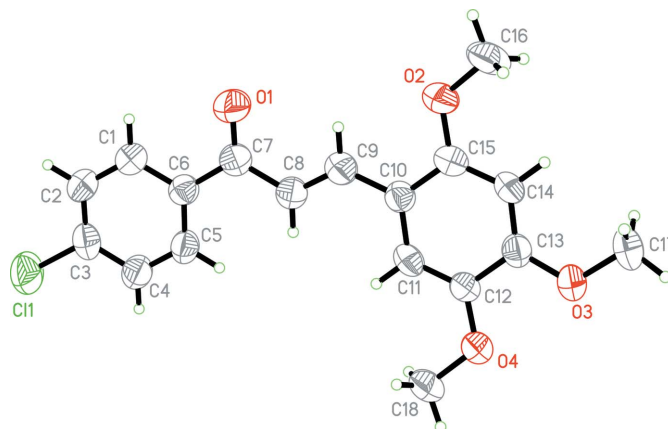
Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atomic numbering.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bosshard, Ch., Sutter, K., Prêtre, Ph., Hulliger, J., Flörsheimer, M., Kaatz, P. & Günter, P. (1995). *Organic Nonlinear Optical Materials. Advances in Nonlinear Optics*, Vol. 1. Amsterdam: Gordon and Breach.
Bruker (2005). *APEX2* (Version 1.27), *SAINT* (Version 7.12A) and *SADABS* (Version 2004/1). Bruker AXS Inc., Madison, Wisconsin, USA.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Goto, Y., Hayashi, A., Kimura, Y. & Nakayama, M. (1991). *J. Cryst. Growth*, **108**, 688–698.
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. & Indira, J. (2006). *Acta Cryst.* E**62**, o1647–o1649.
Harrison, W. T. A., Yathirajan, H. S., Sarojini, B. K., Narayana, B. & Vijaya Raj, K. K. (2006). *Acta Cryst.* E**62**, o1578–o1579.
Nalwa, H. S. & Miyata, S. (1997). *Nonlinear Optics of Organic Molucules and Polymers*. Boca Raton, USA: CRC Press.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Ng, S.-L., Razak, I. A., Fun, H.-K., Patil, P. S., Dharmaprakash, S. M. & Shettigar, V. (2006). *Acta Cryst.* E**62**, o2611–o2613.
Patil, P. S., Teh, J. B.-J., Fun, H.-K., Razak, I. A. & Dharmaprakash, S. M. (2006). *Acta Cryst.* E**62**, o3096–o3098.
Sheldrick, G. M. (1998). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Uchida, T., Kozawa, K., Sakai, T., Aoki, M., Yoguchi, H., Abdureyim, A. & Watanabe, Y. (1998). *Mol. Cryst. Liq. Cryst.* **314**, 135–140.
Zhang, G., Kinoshita, T., Sasaki, K., Goto, Y. & Nakayam, M. (1990). *J. Cryst. Growth*, **100**, 411–416.