

3-(4-Methoxyphenyl)-1-(4-nitrophenyl)prop-2-en-1-one

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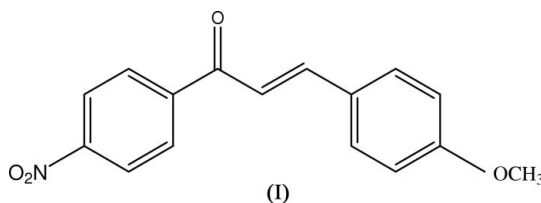
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Key indicators

Single-crystal X-ray study
 $T = 100$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.035
 wR factor = 0.096
Data-to-parameter ratio = 11.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.The enone group and the benzene rings of the title compound, $\text{C}_{16}\text{H}_{13}\text{NO}_4$, are each planar. In the crystal structure, intermolecular $\text{C}-\text{H}\cdots\text{O}$ interactions form chains along the a axis.Received 25 January 2006
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Comment

In the last two decades, much effort has been focused on the discovery of new organic materials which exhibit large non-linear optical (NLO) properties (Chemla & Zyss, 1987) and would thus have applications in the field of opto-electronics and photonics. In order to obtain second-order NLO single crystals, the main requirements are the choice of molecules with a large hyperpolarizability (β) and the alignment of these molecules in a non-centrosymmetric space group with optimal orientation in the crystal structure. Among the many known organic NLO materials, chalcone derivatives are interesting as they exhibit extremely high and fast non-linearity (Fichou *et al.*, 1988; Uchida *et al.*, 1998; Goto *et al.*, 1991; Zhao *et al.*, 2000) and show a preference to crystallize as non-centrosymmetric structures. In this connection we synthesized the title compound, (I), as a potential second-order NLO material and, in order to obtain detailed information on its crystal structure, an X-ray crystal structure determination of (I) has been carried out.



The non-centrosymmetric space group of (I) is consistent with the non-zero SHG signal observed. Our measurements of the SHG conversion efficiency of (I) show that it is five times that of urea.

A molecular view of (I) is shown in Fig. 1. Bond lengths and angles have normal values (Allen *et al.*, 1987) and are comparable to those in related structures (Sathiyamoorthi, Chinnakali, Nanjundan, Santhi *et al.*, 2005; Sathiyamoorthi, Chinnakali, Nanjundan, Unnithan *et al.*, 2005; Sathiyamoorthi, Chinnakali, Nanjundan, Radhika *et al.*, 2005; Ravishankar *et al.*, 2005; Teh *et al.*, 2006; Ng *et al.*, 2006). The enone group and the benzene rings of the chalcone are each planar. The least-squares plane through the enone group makes dihedral angles of $10.1(1)^\circ$ and $8.0(1)^\circ$ with the C1–C6 and C10–C15 benzene rings, respectively. The dihedral angle between the benzene rings is $12.06(5)^\circ$. The nitro group

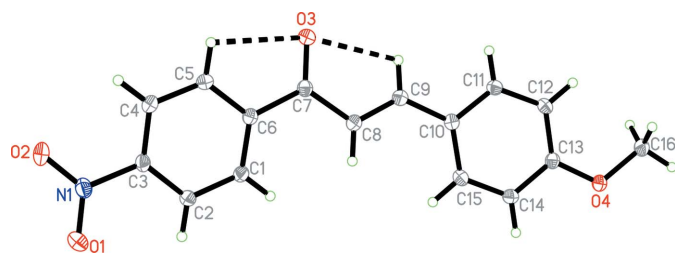


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atomic numbering. Dashed lines represent hydrogen bonds.

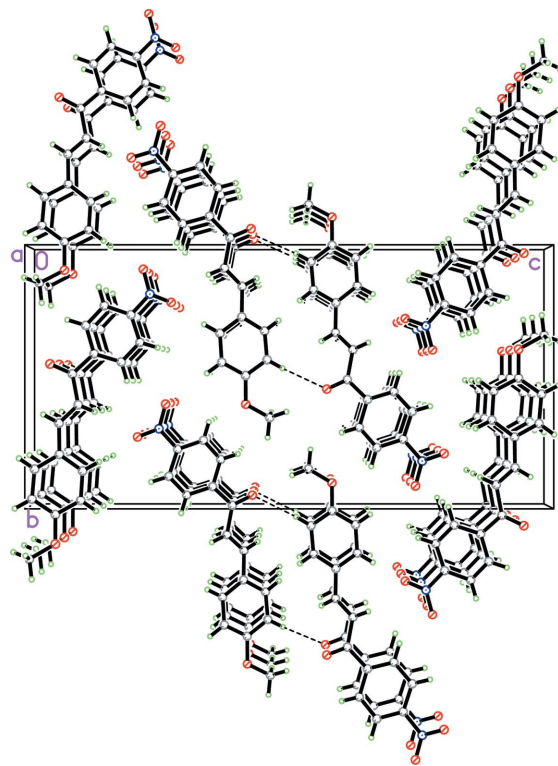


Figure 2
The crystal packing of (I), viewed down the *a* axis. Hydrogen bonds are shown as dashed lines.

attached at C3 is twisted away from the C1–C6 benzene ring, with torsion angles O1–N1–C3–C2 of 10.9 (2)° and O2–N1–C3–C4 of 11.7 (2)°. Meanwhile, the methoxy group attached at C13 is coplanar with the C10–C15 benzene ring.

In the molecular structure of (I), each of the C5–H5A···O3 and C9–H9A···O3 interactions generates an S(5) ring motif (Bernstein *et al.*, 1995). The crystal structure is stabilized by C–H···O interactions (Table 1), which form molecular chains along the *a* axis (Fig. 2).

Experimental

Compound (I) was prepared by the condensation of 4-methoxybenzaldehyde (0.01 mol) with 4-nitroacetophenone (0.01 mol) in ethanol (60 ml) in the presence of NaOH (5 ml, 30%). After stirring for 2 h, the contents of the flask were poured into ice-cold water, and the resulting crude solid was collected by filtration. The compound was dried and recrystallized from acetone. Crystals suitable for X-ray

diffraction study were grown by slow evaporation of an acetone solution. The SHG measurements were carried out by the classical powder technique (Kurtz & Perry, 1968) using a pulsed Nd:YAG laser (1.064 μm, 10 ns, 5 mJ).

Crystal data

C₁₆H₁₃NO₄
M_r = 283.27
 Orthorhombic, *P*2₁2₁2₁
a = 3.8765 (1) Å
b = 12.9341 (2) Å
c = 25.9060 (5) Å
V = 1298.90 (5) Å³
Z = 4
D_x = 1.449 Mg m^{−3}

Mo *K*α radiation
 Cell parameters from 9254 reflections
 θ = 1.8–30.0°
 μ = 0.11 mm^{−1}
T = 100.0 (1) K
 Block, yellow
 0.79 × 0.42 × 0.17 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 2005)
T_{min} = 0.813, *T_{max}* = 0.982
 15897 measured reflections

2247 independent reflections
 2164 reflections with *I* > 2σ(*I*)
R_{int} = 0.028
 θ_{\max} = 30.0°
h = −5 → 5
k = −18 → 18
l = −35 → 35

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.035
wR(*F*²) = 0.096
S = 1.06
 2247 reflections
 190 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0585P)^2 + 3.3408P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.32 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.22 \text{ e \AA}^{-3}$

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
C5–H5···O3	0.93	2.42	2.746 (2)	100
C9–H9···O3	0.93	2.41	2.781 (2)	104
C12–H12···O3 ⁱ	0.93	2.54	3.470 (2)	175

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

H atoms were placed in calculated positions, with C–H distances of 0.93 or 0.96 Å. The *U*_{iso}(H) values were constrained to be 1.5*U*_{eq} of the carrier atom for methyl H atoms and 1.2*U*_{eq} for the remaining H atoms. In the absence of significant anomalous dispersion effects, Friedel pairs were merged before the final refinement.

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).

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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. S1–19.

- Bernstein, J., Davis, R. E., Shimoni, L. & Chang N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
- Bruker (2005). *APEX2* (Version 1.27), *SAINTE* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Chemla, D. S. & Zyss, J. (1987). *Nonlinear Optical Properties of Organic Molecules & Crystals*, Vol. 1 and 2. New York: Academic Press.
- Fichou, D., Watanabe, T., Takeda, T., Miyata, S., Goto, Y. & Nakayama, M. (1988). *Jpn J. Appl. Phys.* **27**, L429–L430.
- Goto, Y., Hayashi, A., Kimura, Y. & Nakayama, M. (1991). *J. Cryst. Growth*, **108**, 688–698.
- Kurtz, S. K. & Perry, T. T. (1968). *J. Appl. Phys.* **39**, 3798–3813.
- Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
- Ng, S. L., Patil, P. S., Razak, I. A., Fun, H. -K. & Dharmaparakash, S. M. (2006). *Acta Cryst.* **E62**, o893–o895.
- Ravishankar, T., Chinnakali, K., Nanjundan, S., Selvam, P., Fun, H.-K. & Yu, X. -L. (2005). *Acta Cryst.* **E61**, o405–o407.
- Sathiya Moorthi, S., Chinnakali, K., Nanjundan, S., Radhika, R., Fun, H.-K. & Yu, X.-L. (2005). *Acta Cryst.* **E61**, o480–o482.
- Sathiya Moorthi, S., Chinnakali, K., Nanjundan, S., Santhi, R. & Fun, H.-K. (2005). *Acta Cryst.* **E61**, o3514–o3516.
- Sathiya Moorthi, S., Chinnakali, K., Nanjundan, S., Unnithan, C. S., Fun, H.-K. & Yu, X.-L. (2005). *Acta Cryst.* **E61**, o483–o485.
- Sheldrick, G. M. (1998). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Teh, J. B. J., Patil, P. S., Fun, H.-K., Razak, I. A., & Dharmaparakash, S. M. (2006). *Acta Cryst.* **E62**, o890–o892.
- Uchida, T., Kozawa, K., Sakai, T., Aoki, M., Yoguchi, H., Abdureyim, A. & Watanebe, Y. (1998). *Mol. Cryst. Liq. Cryst.* **314**, 135–140.
- Zhao, B., Lu, W.-Q., Zhou, Z.-H. & Wu, Y. (2000). *J. Mater. Chem.* **10**, 1513–1517.