

A redetermination of 1,5-bis(4-methoxyphenyl)penta-1,4-dien-3-one at 120 (2) K

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Key indicators

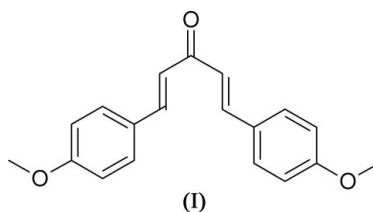
Single-crystal X-ray study
 T = 120 K
 Mean $\sigma(\text{C}-\text{C}) = 0.005 \text{ \AA}$
 R factor = 0.052
 wR factor = 0.127
 Data-to-parameter ratio = 9.2

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

The title compound, $\text{C}_{19}\text{H}_{18}\text{O}_3$, is confirmed to crystallize with orthorhombic symmetry [Shan *et al.* (1999). *Z. Kristallogr. New Cryst. Struct.* **214**, 381–382; Marsh (2004). *Acta Cryst. B* **60**, 252–253]. The molecule has crystallographically imposed twofold symmetry and the only possible intermolecular interaction is a weak $\text{C}-\text{H}\cdots\text{O}$ bond.

Comment

Although the title compound (common name bischalcone), (I), was first prepared over 100 years ago (von Baeyer & Villiger, 1902), it was not until 1999 that its single-crystal structure was determined (Shan *et al.*, 1999). These workers described its structure as monoclinic (space group *Cc*), with all atoms occupying general positions. Later, Marsh (2004) noted that the crystal symmetry of (I) is better described as orthorhombic (space group *Aba2*), as confirmed by the present study. This compound is of interest to us on account of its substantial second harmonic generation (SHG) response (six times that of urea) to red light. The current structure determination at 120 (2) K represents a significant improvement in precision compared with the structure determined from room-temperature data.



The geometric parameters for (I) are normal. The complete molecule is generated from the asymmetric unit by twofold symmetry, with atoms C1 and O1 lying on the rotation axis (Fig. 1). The dihedral angle between the two benzene rings in (I) is $56.92(9)^\circ$. A short $\text{H}3\cdots\text{H}3^i$ [symmetry code: (i) $1 - x, 1 - y, z$] intramolecular contact of 2.18 \AA is present, which may help to explain the twisted conformation of the molecule about the central ketone group [pseudo-torsion angle $\text{C}3-\text{C}2\cdots\text{C}2^i-\text{C}3^i = -45.1(4)^\circ$]. Atoms C3, O2 and C10 are displaced from the mean plane of the C4–C9 benzene ring by $0.111(5)$, $0.024(4)$ and $0.128(6) \text{ \AA}$, respectively.

The packing in (I), shown in Fig. 2, results in head-to-tail columns of molecules which all propagate along [001] in the same sense; the large SHG signal of (I) could be correlated with this lining-up effect. A *PLATON* (Spek, 2003) analysis of (I) identified a possible $\text{C}-\text{H}\cdots\text{O}$ interaction (Table 1) that might help to crosslink these molecular columns. There are no $\pi-\pi$ stacking interactions in (I).

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Experimental

Compound (I) was prepared according to a literature method (Vogel, 1999) and recrystallized from acetone by slow evaporation (m.p. 378–381 K). Elemental analysis, found: C 77.25, H 6.02%; calculated for $C_{19}H_{18}O_3$: C 77.55, H 6.12%.

Crystal data

$C_{19}H_{18}O_3$
 $M_r = 294.33$
 Orthorhombic, *Aba2*
 $a = 7.2756$ (9) Å
 $b = 33.5830$ (6) Å
 $c = 6.132$ (5) Å
 $V = 1498.3$ (12) Å³
 $Z = 4$
 $D_x = 1.305$ Mg m⁻³

Mo $K\alpha$ radiation
 Cell parameters from 1686 reflections
 $\theta = 1.0$ – 27.5°
 $\mu = 0.09$ mm⁻¹
 $T = 120$ (2) K
 Lath, pale yellow
 $0.52 \times 0.22 \times 0.04$ mm

Data collection

Bruker-Nonius KappaCCD diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 2003)
 $T_{\min} = 0.956$, $T_{\max} = 0.997$
 8103 measured reflections

942 independent reflections
 632 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.093$
 $\theta_{\text{max}} = 27.8^\circ$
 $h = -9 \rightarrow 8$
 $k = -43 \rightarrow 43$
 $l = -6 \rightarrow 7$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.052$
 $wR(F^2) = 0.127$
 $S = 1.10$
 942 reflections
 102 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0604P)^2 + 0.238P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.22$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.24$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C10-H10A\cdots O2^i$	0.98	2.53	3.442 (4)	154

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

In the absence of significant anomalous scattering effects, Friedel pairs were merged prior to refinement. The H atoms were positioned geometrically (C–H = 0.95–0.98 Å) and refined as riding, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier})$ or $1.5U_{\text{eq}}(\text{methyl carrier})$. The methyl group was rotated to fit the electron density.

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

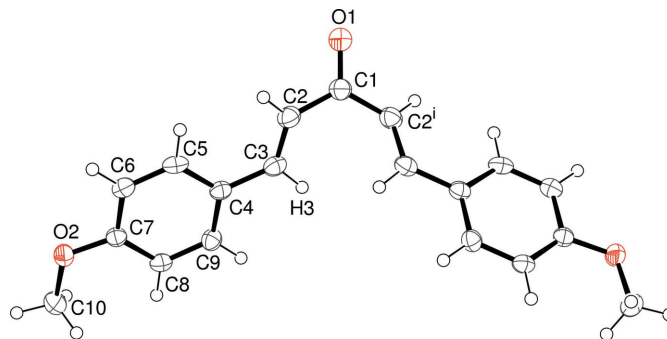


Figure 1

View of (I), showing 50% displacement ellipsoids, with arbitrary spheres for H atoms. [Symmetry code: (i) $1 - x, 1 - y, z$].

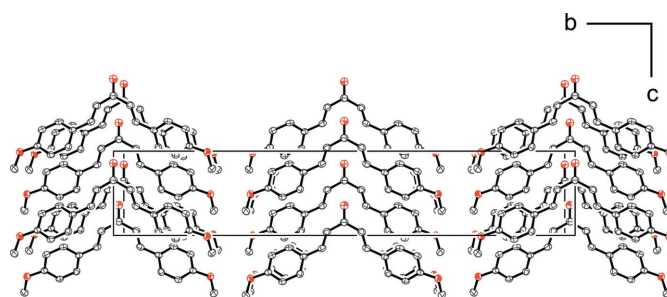


Figure 2

The molecular packing in (I), viewed along [100], with H atoms omitted for clarity.

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