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

William T. A. Harrison,[a]* B. K. Sarojini,[b] K. K. Vijaya Raj,[c] H. S. Yathirajan[d] and B. Narayana[c]

[a]Department of Chemistry, University of Aberdeen, Meston Walk, Aberdeen AB24 3UE, Scotland, [b]Department of Chemistry, P. A. College of Engineering, Nadupadavu, Mangalore 574 153, India, [c]Department of Chemistry, Mangalore University, Mangalagangotri 574 199, India, and [d]Department of Studies in Chemistry, University of Mysore, Manasagangotri, Mysore 570 006, India

Correspondence e-mail: w.harrison@abdn.ac.uk

Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.005 Å
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.

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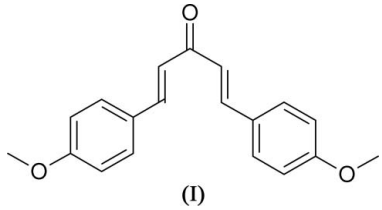
The title compound, $C_{19}H_{18}O_3$, is confirmed to crystallize with orthorhombic symmetry [Shan *et al.* (1999). *Z. Kristallogr. New Cryst. Struct.* **214**, 381–382; Marsh (2004). *Acta Cryst.* B**60**, 252–253]. The molecule has crystallographically imposed twofold symmetry and the only possible intermolecular interaction is a weak C—H···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 *Aba*2), 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.



(I)

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)°. A short H3···H3^{i} [symmetry code: (i) $1-x$, $1-y$, z] intramolecular contact of 2.18 Å is present, which may help to explain the twisted conformation of the molecule about the central ketone group [pseudo-torsion angle C3—C2···C2^{i}—C3^{i} = −45.1 (4)°]. 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) Å, 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 C—H···O interaction (Table 1) that might help to crosslink these molecular columns. There are no π–π stacking interactions in (I).

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, *Aba*2
a = 7.2756 (9) Å
b = 33.5830 (6) Å
c = 6.132 (5) Å
V = 1498.3 (12) Å^3
Z = 4
D_x = 1.305 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1686 reflections
θ = 1.0–27.5°
μ = 0.09 mm^{-1}
T = 120 (2) K
Lath, pale yellow
0.52 × 0.22 × 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_{int} = 0.093
θ_{max} = 27.8°
$h = -9 \rightarrow 8$
$k = -43 \rightarrow 43$
$l = -6 \rightarrow 7$

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 $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C10—H10A···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_{iso}(H) = 1.2U_{eq}(carrier) or 1.5U_{eq}(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*.

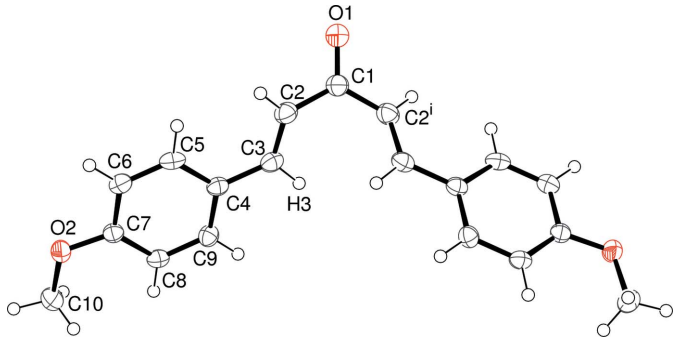


Figure 1
View of (I), showing 50% displacement ellipsoids, with arbitrary spheres for H atoms. [Symmetry code: (i) $1-x, 1-y, z$.]

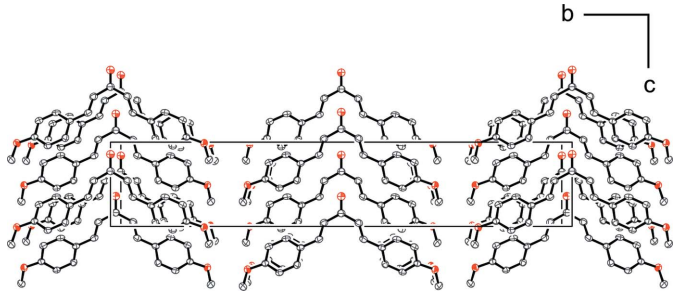


Figure 2
The molecular packing in (I), viewed along [100], with H atoms omitted for clarity.

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