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

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Key indicators

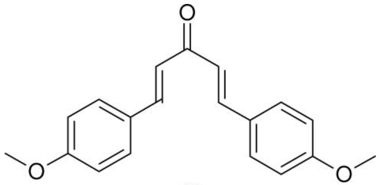
Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.005 Å
R factor = 0.052
wR factor = 0.127
Data-to-parameter ratio = 9.2

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

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
$\theta = 1.0$–$27.5°$
$\mu = 0.09$ 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 $I > 2\sigma(I)$
$R_{\rm int} = 0.093$
$\theta_{\max} = 27.8°$
$h = -9 \rightarrow 8$
$k = -43 \rightarrow 43$
$l = -6 \rightarrow 7$

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$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C10—H10$A\cdots$O2$^{\rm i}$	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 $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(carrier) or $1.5U_{\rm eq}$(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*.

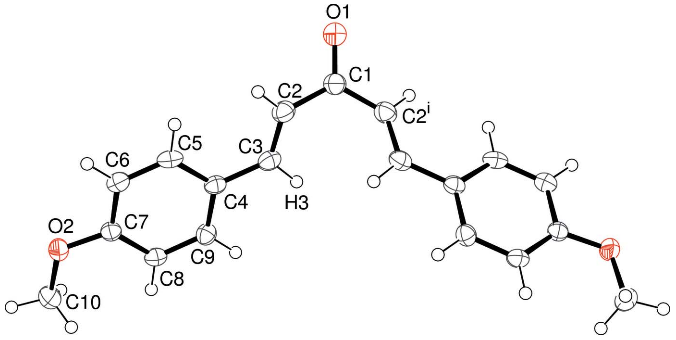


Figure 1
View of (I), showing 50% displacement ellipsoids, with arbitrary spheres for H atoms. [Symmetry code: (i) $1 - x, 1 - y, z$.]

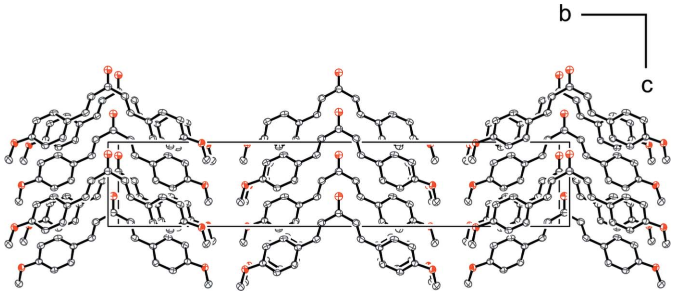


Figure 2
The molecular packing in (I), viewed along [100], with H atoms omitted for clarity.

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supporting information

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Crystal data

$C_{19}H_{18}O_3$
$M_r = 294.33$
Orthorhombic, *Aba*2
Hall symbol: A 2 -2ac
$a = 7.2756$ (9) Å
$b = 33.5830$ (6) Å
$c = 6.132$ (5) Å
$V = 1498.3$ (12) Å^3
$Z = 4$
$F(000) = 624$
$D_x = 1.305$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1686 reflections
$\theta = 1.0$–27.5°
$\mu = 0.09$ mm^{-1}
$T = 120$ K
Lath, pale yellow
0.52 × 0.22 × 0.04 mm

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Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
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942 independent reflections
632 reflections with $I > 2\sigma(I)$
$R_{int} = 0.093$
$\theta_{max} = 27.8°$, $\theta_{min} = 3.1°$
$h = -9 \rightarrow 8$
$k = -43 \rightarrow 43$
$l = -6 \rightarrow 7$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.127$
$S = 1.10$
942 reflections
102 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: none
Hydrogen site location: inferred from neighbouring sites
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}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	$U_{\rm iso}$*/$U_{\rm eq}$
C1	0.5000	0.5000	0.3446 (9)	0.0326 (13)
C2	0.4564 (4)	0.46240 (9)	0.4579 (6)	0.0331 (9)
H2	0.3791	0.4441	0.3832	0.040*
C3	0.5145 (5)	0.45178 (10)	0.6529 (6)	0.0302 (9)
H3	0.5728	0.4717	0.7381	0.036*
C4	0.4981 (4)	0.41190 (9)	0.7517 (6)	0.0262 (8)
C5	0.4102 (4)	0.38001 (10)	0.6492 (6)	0.0299 (8)
H5	0.3505	0.3842	0.5134	0.036*
C6	0.4086 (4)	0.34238 (9)	0.7415 (6)	0.0288 (8)
H6	0.3473	0.3212	0.6690	0.035*
C7	0.4957 (4)	0.33534 (10)	0.9389 (5)	0.0262 (8)
C8	0.5818 (4)	0.36670 (9)	1.0448 (6)	0.0270 (8)
H8	0.6417	0.3624	1.1804	0.032*
C9	0.5801 (4)	0.40439 (9)	0.9520 (5)	0.0284 (8)
H9	0.6369	0.4257	1.0280	0.034*
C10	0.5861 (5)	0.28785 (10)	1.2094 (5)	0.0413 (10)
H10A	0.5660	0.2599	1.2480	0.062*
H10B	0.5414	0.3049	1.3277	0.062*
H10C	0.7177	0.2925	1.1870	0.062*
O1	0.5000	0.5000	0.1441 (6)	0.0465 (11)
O2	0.4879 (3)	0.29697 (6)	1.0122 (5)	0.0323 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.036 (3)	0.033 (3)	0.029 (4)	0.003 (2)	0.000	0.000
C2	0.0354 (18)	0.034 (2)	0.030 (2)	0.0011 (15)	−0.0052 (19)	−0.0039 (18)
C3	0.0305 (19)	0.034 (2)	0.026 (2)	0.0038 (15)	0.0008 (19)	−0.0055 (17)
C4	0.0258 (16)	0.0304 (17)	0.0225 (18)	0.0024 (15)	0.0025 (17)	−0.0029 (16)
C5	0.0272 (19)	0.043 (2)	0.0193 (15)	0.0021 (15)	−0.0021 (16)	−0.0035 (17)
C6	0.0275 (17)	0.0335 (18)	0.0254 (18)	−0.0029 (15)	0.0008 (18)	−0.0045 (17)
C7	0.0273 (16)	0.0272 (16)	0.024 (2)	0.0012 (14)	0.0050 (19)	−0.0007 (14)
C8	0.0266 (17)	0.0331 (18)	0.0212 (17)	0.0019 (15)	−0.0022 (15)	−0.0016 (16)
C9	0.0273 (16)	0.0267 (17)	0.031 (2)	0.0017 (14)	−0.0018 (19)	−0.0024 (18)
C10	0.057 (2)	0.032 (2)	0.034 (2)	0.0021 (18)	−0.009 (2)	0.0054 (19)
O1	0.071 (3)	0.034 (2)	0.034 (2)	−0.0016 (18)	0.000	0.000
O2	0.0397 (13)	0.0251 (11)	0.0320 (14)	−0.0027 (11)	−0.0040 (13)	0.0013 (11)

Geometric parameters (Å, º)

C1—O1	1.230 (6)	C6—C7	1.386 (5)
C1—C2^{i}	1.476 (4)	C6—H6	0.9500
C1—C2	1.476 (4)	C7—O2	1.366 (4)
C2—C3	1.318 (5)	C7—C8	1.387 (4)
C2—H2	0.9500	C8—C9	1.388 (4)
C3—C4	1.475 (4)	C8—H8	0.9500
C3—H3	0.9500	C9—H9	0.9500
C4—C9	1.389 (5)	C10—O2	1.438 (4)
C4—C5	1.397 (5)	C10—H10A	0.9800
C5—C6	1.385 (4)	C10—H10B	0.9800
C5—H5	0.9500	C10—H10C	0.9800
O1—C1—C2^{i}	118.1 (2)	C7—C6—H6	119.7
O1—C1—C2	118.1 (2)	O2—C7—C6	115.4 (3)
C2^{i}—C1—C2	123.8 (5)	O2—C7—C8	125.5 (3)
C3—C2—C1	126.1 (3)	C6—C7—C8	119.1 (3)
C3—C2—H2	116.9	C7—C8—C9	119.8 (3)
C1—C2—H2	116.9	C7—C8—H8	120.1
C2—C3—C4	126.4 (4)	C9—C8—H8	120.1
C2—C3—H3	116.8	C8—C9—C4	122.2 (3)
C4—C3—H3	116.8	C8—C9—H9	118.9
C9—C4—C5	117.1 (3)	C4—C9—H9	118.9
C9—C4—C3	119.6 (3)	O2—C10—H10A	109.5
C5—C4—C3	123.3 (4)	O2—C10—H10B	109.5
C6—C5—C4	121.3 (3)	H10A—C10—H10B	109.5
C6—C5—H5	119.4	O2—C10—H10C	109.5
C4—C5—H5	119.4	H10A—C10—H10C	109.5
C5—C6—C7	120.6 (3)	H10B—C10—H10C	109.5
C5—C6—H6	119.7	C7—O2—C10	117.2 (2)
O1—C1—C2—C3	152.1 (3)	C5—C6—C7—C8	1.2 (4)
C2^{i}—C1—C2—C3	−27.9 (3)	O2—C7—C8—C9	179.8 (3)
C1—C2—C3—C4	−168.9 (3)	C6—C7—C8—C9	−0.2 (5)
C2—C3—C4—C9	174.6 (3)	C7—C8—C9—C4	−1.6 (5)
C2—C3—C4—C5	−2.3 (5)	C5—C4—C9—C8	2.3 (4)
C9—C4—C5—C6	−1.3 (5)	C3—C4—C9—C8	−174.8 (3)
C3—C4—C5—C6	175.6 (3)	C6—C7—O2—C10	176.3 (3)
C4—C5—C6—C7	−0.4 (5)	C8—C7—O2—C10	−3.8 (4)
C5—C6—C7—O2	−178.9 (3)		

Symmetry code: (i) $-x+1, -y+1, z$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

C10—H10*A*···O2^{ii}	0.98	2.53	3.442 (4)	154

Symmetry code: (ii) −*x*+1, −*y*+1/2, *z*+1/2.