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3,3′-(*p*-Phenylenedimethylene)bis(pentane-2,4-dione)

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Key indicators

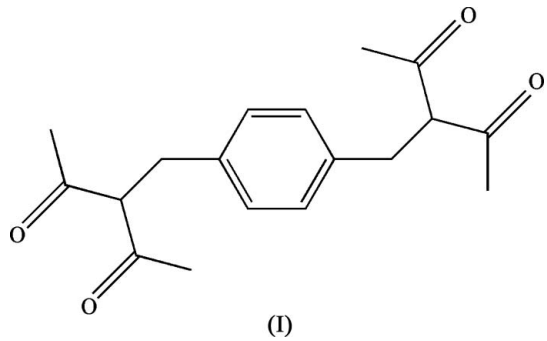
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.065
wR factor = 0.157
Data-to-parameter ratio = 16.4

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

The title molecule, $C_{18}H_{22}O_4$, lies on a crystallographic inversion center which is located at the center of the benzene ring. In the crystal structure, moleclues are linked by weak intermolecular C—H···O hydrogen bonds, forming a two-dimensional network perpendicular to the *a* axis.

Comment

The title compound, (I), was originally synthesized by Martin *et al.* (1959), but its crystal structure has not hitherto been reported. The presence of two β-diketone groups suggests that (I) may be a useful bridging ligand. The molecular structure of (I) is shown in Fig. 1. The molecule lies on a crystallographic inversion center located at the center of the benzene ring.



(I)

In the crystal structure, weak intermolecular C—H···O hydrogen bonds link molecules into a two-dimensional network perpendicular to the *a* axis (Table 1 and Fig. 2)

Experimental

The title compound was synthesized according to Martin *et al.* (1959). To a solution of potassium (3.9 g, 0.1 mol) in 2-methyl-2-propanol (100 ml), acetylacetone (10.0 g, 0.1 mol) was added dropwise and the

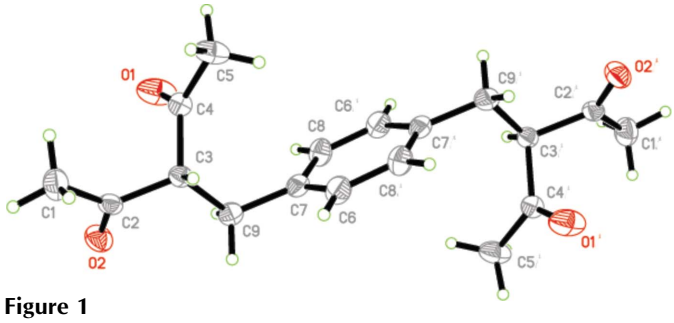


Figure 1
The molecular structure of (I), showing the atom-numbering scheme, with displacement ellipsoids drawn at the 30% probability level. [Symmetry code: (i) $-x$, $-y$, $-z+1$.]

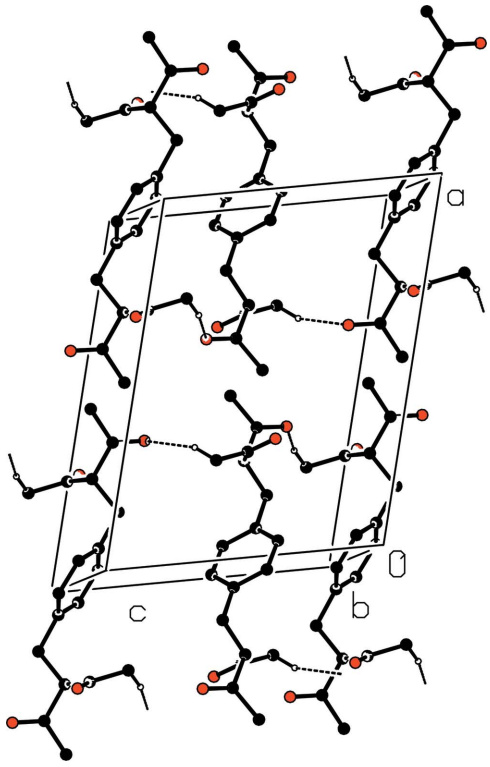


Figure 2
A packing plot (Spek, 2003), showing hydrogen bonds as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

mixture was refluxed for 30 min with stirring. 1,4-Dibromobenzene (13.2 g, 0.05 mol) was then added over a period of 40 min and KI (2.0 g, 0.012 mol) was then added. The mixture was stirred and refluxed for 4 h, and then 75% of the solvent was distilled and the solid residue was washed thoroughly with H_2O and dried. Single crystals were obtained by recrystallization from H_2O/acetonitrile in a 2:1 ratio. Elemental analysis found: C 71.72, H 7.15%; calculated for $C_{18}H_{22}O_4$: C 71.50, H 7.33%. The IR spectrum reveals a strong peak at 1724 cm^{-1} for the vibration of carbonyl groups.

Crystal data

$C_{18}H_{22}O_4$
M_r = 302.36
Monoclinic, $P2_1/c$
a = 13.959 (5) Å
b = 5.679 (2) Å
c = 10.860 (4) Å
β = 107.386 (6)°
V = 821.6 (5) Å^3
Z = 2
D_x = 1.222 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 (2) K
Prism, colorless
0.30 × 0.21 × 0.12 mm

Data collection

Bruker SMART APEX CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.975, T_{max} = 0.990
4365 measured reflections
1669 independent reflections
1189 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 26.4°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.157
S = 1.08
1669 reflections
102 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0555P)^2 + 0.239P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H7C···O2^{i}	0.96	2.53	3.385 (4)	149

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

H atoms were placed in calculated positions and refined as riding, with benzene C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C), methyl C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C), methylene C—H = 0.97 Å and U_{iso}(H) = 1.2U_{eq}(C), methine C—H = 0.98 Å and U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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