Related literature. A search of the most recent Cambridge Structural Database file did not produce any similar structures.

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Structure of (*S*,*S*)-3,6-Diphenyl-1,4-dioxane-2,5-dione

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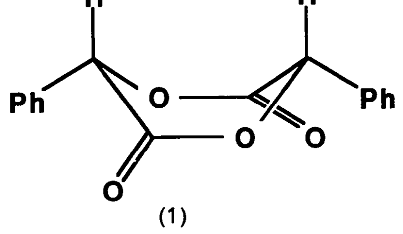
Abstract. $C_{16}H_{12}O_4$, $M_r = 268{\cdot}27$, orthorhombic, $P2_12_12_1$, $a = 5{\cdot}103\,(3)$, $b = 9{\cdot}272\,(6)$, $c = 26{\cdot}50\,(3)$ Å, $V = 1254\,(2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}42$ g cm^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}7107$ Å, $\mu = 0{\cdot}9574$ cm^{-1}, $F(000) = 560$, $T =$ 198 K, $R = 0{\cdot}0542$ for 886 reflections $[F_o \geq 4\sigma(F_o)]$. The absolute configuration is assigned on the basis of internal comparison to the (+)-mandelate moiety. Close C—H···O contacts are observed resulting in a two-dimensional network of H-bonded molecules lying parallel to *ab*. Atom H3 is involved in a three-center C—H···O-type hydrogen bond. The shorter H···O contact involves C3—H3···O8 ($1 - x$, $\frac{1}{2} + y$, $\frac{3}{2} - z$) with H3···O8 2·26 (6) Å, C3···O8 2·949 (7) Å and C—H···O 127 (4)°, while the longer contact involves C3—H3···O7 ($x - 1$, y, z) with H3···O7 2·41 (6) Å, C3···O7 3·143 (8) Å and C—H···O 132 (4)°.

Experimental. (1) was prepared by *p*-toluenesulfonic acid catalyzed self-condensation of *S*-(+)-mandelic acid in benzene at reflux with azeotropic removal of water (Pojman, 1988). After recrystallization from tetrahydrofuran, the material was sublimed *in vacuo* providing single crystals suitable for X-ray analysis. The data crystal was a colorless needle of approximate dimensions 0·03 × 0·13 × 0·54 mm. The data were collected on a Nicolet *R*3 diffractometer using a graphite monochromator and equipped with a Nicolet LT-2 low-temperature assembly. Lattice parameters were obtained from the least-squares refinement of 36 reflections with $12{\cdot}7 < 2\theta < 21{\cdot}3°$. The data were collected using the ω-scan technique (2444 reflections, of which 1323 were unique, $R_{\rm int} = 0{\cdot}0412$ from averaging hkl and $h\bar{k}\bar{l}$ reflections), with a 2θ range from 4·0–50·0°, and a 1·0° ω scan at a scan rate of 1–4° min^{-1} ($h = 0 \rightarrow 6$, $k = -11 \rightarrow 11$, $l = -31 \rightarrow 31$). Four reflections ($00\bar{6}$; $\bar{1}\bar{1}0$; $1\bar{2}3$; 021) were remeasured every 96 reflections to monitor instrument and crystal stability (maximum correction on I was < 4%). The data were also corrected for Lp effects but not for absorption. The decay correction and the data reduction were performed using *SHELXTL-PLUS* (Sheldrick, 1987). Reflections having $F_o < 4\sigma(F_o)$ were considered unobserved (437 reflections). The structure was solved by direct methods (Sheldrick, 1987) and refined by full-matrix least squares (Sheldrick, 1976). The non-H atoms were refined with anisotropic thermal parameters. All H-atom positions were obtained from a ΔF map and refined with isotropic thermal parameters. After several cycles of refinement, the $U_{\rm iso}$ for H3 refined to a negative value and, therefore, was fixed at 1·2 × $U_{\rm eq}$ of C3 for the final refinement cycles. In all, 228 parameters were refined. The function $\sum w(|F_o| - |F_c|)^2$ was minimized, where $w = 1/[\sigma(F_o)]^2$ and $\sigma(F_o) = 0{\cdot}5kI^{-1/2}\{[\sigma(I)]^2 + (0{\cdot}02I)^2\}^{1/2}$. The intensity I is given by $(I_{\rm peak} - I_{\rm background}) \times$ (scan rate); 0·02 is a factor to downweight intense reflections and to account for instrument instability and k is the correction due to Lp effects and decay. $\sigma(I)$ was estimated from counting statistics; $\sigma(I) = [(I_{\rm peak} + I_{\rm background})^{1/2} \times$ (scan rate)]. The final $R = 0{\cdot}0542$ for 886 reflections, with $wR = 0{\cdot}0487$ ($R_{\rm all} = 0{\cdot}0919$, $wR_{\rm all} = 0{\cdot}0557$) and goodness of fit = 1·250. The maximum $|\Delta/\sigma| < 0{\cdot}1$ in the final refinement cycle and the



(1)

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Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors*

$U_{\rm eq} = (1/3)\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	$U_{\rm eq}$(Å^2)
O1	0·8855 (9)	0·9598 (4)	0·72154 (13)	0·0208 (13)
C2	0·9426 (13)	0·9720 (6)	0·7711 (2)	0·021 (2)
C3	0·7325 (13)	0·9227 (6)	0·8062 (2)	0·020 (2)
O4	0·6053 (9)	0·7955 (4)	0·78614 (12)	0·0205 (13)
C5	0·5061 (14)	0·8069 (6)	0·7395 (2)	0·020 (2)
C6	0·6191 (13)	0·9295 (6)	0·7084 (2)	0·020 (2)
O7	1·1523 (9)	1·0149 (4)	0·78302 (14)	0·0260 (14)
O8	0·3439 (9)	0·7254 (4)	0·72465 (14)	0·0251 (14)
C9	0·8254 (12)	0·8864 (6)	0·8582 (2)	0·020 (2)
C10	0·7120 (14)	0·9551 (7)	0·8981 (2)	0·030 (2)
C11	0·794 (2)	0·9201 (9)	0·9468 (2)	0·048 (3)
C12	0·985 (2)	0·8225 (9)	0·9555 (3)	0·041 (3)
C13	1·098 (2)	0·7538 (9)	0·9148 (3)	0·043 (3)
C14	1·0168 (14)	0·7861 (7)	0·8655 (2)	0·032 (2)
C15	0·6140 (13)	0·8936 (6)	0·6528 (2)	0·020 (2)
C16	0·785 (2)	0·7964 (7)	0·6336 (2)	0·031 (2)
C17	0·773 (2)	0·7585 (8)	0·5831 (2)	0·036 (2)
C18	0·586 (2)	0·8210 (9)	0·5525 (2)	0·044 (3)
C19	0·415 (2)	0·9158 (10)	0·5719 (2)	0·046 (3)
C20	0·426 (2)	0·9543 (7)	0·6233 (2)	0·034 (2)

Table 2. *Bond lengths* (Å) *and angles* (°)

1	2	3	1—2	1—2—3
C2	O1	C6	1·350 (7)	117·3 (5)
C6	O1		1·431 (8)	
C3	C2	O7	1·491 (9)	125·9 (5)
C3	C2	O1		115·2 (5)
O7	C2	O1	1·185 (8)	118·8 (5)
O4	C3	C9	1·447 (7)	107·3 (4)
O4	C3	C2		110·1 (4)
C9	C3	C2	1·495 (8)	114·5 (5)
C5	O4	C3	1·339 (7)	116·4 (4)
C6	C5	O8	1·519 (8)	124·1 (5)
C6	C5	O4		114·6 (5)
O8	C5	O4	1·188 (8)	121·3 (5)
C15	C6	O1	1·509 (8)	107·3 (5)
C15	C6	C5		111·0 (5)
O1	C6	C5		112·0 (5)
C10	C9	C14	1·364 (8)	120·8 (5)
C10	C9	C3		118·4 (5)
C14	C9	C3	1·362 (9)	120·8 (5)
C11	C10	C9	1·394 (9)	118·8 (7)
C12	C11	C10	1·348 (12)	122·1 (7)
C13	C12	C11	1·381 (11)	118·6 (7)
C14	C13	C12	1·403 (10)	120·2 (7)
C9	C14	C13		119·5 (6)
C16	C15	C20	1·354 (9)	120·9 (6)
C16	C15	C6		120·2 (5)
C20	C15	C6	1·361 (9)	118·8 (6)
C17	C16	C15	1·386 (9)	120·3 (6)
C18	C17	C16	1·379 (11)	119·4 (7)
C19	C18	C17	1·341 (12)	120·1 (6)
C20	C19	C18	1·408 (9)	120·7 (8)
C15	C20	C19		118·6 (7)

minimum and maximum peaks in the final ΔF map were $-0{\cdot}31$ and $0{\cdot}26\,{\rm e\,\AA^{-3}}$, respectively. The enantiomorph could not be distinguished on the basis of the X-ray diffraction results (wR for the enantiomorph = 0·0487). The scattering factors for the non-H atoms were taken from Cromer & Mann (1968), with the anomalous-dispersion corrections taken from Cromer & Liberman (1970). The scattering factors for the H atoms were obtained from Stewart, Davidson & Simpson (1965). Values used to calculate the linear absorption coefficient are from *International Tables for X-ray Crystallography* (1974). Figures were generated using *SHELXTL-PLUS* (Sheldrick, 1987). The positional and thermal parameters for non-H atoms are listed in Table 1, while the bond lengths and angles for the non-H atoms are listed in Table 2.* The atomic labeling scheme is shown in Fig. 1 and a unit-cell packing diagram is shown in Fig. 2. Other computer programs used in this work are listed in reference 11 of Gadol & Davis (1982).

Related literature. This cyclic dimer of mandelic acid was obtained as a byproduct from the acid catalyzed polymerization of mandelic acid. The single-crystal structure determination was undertaken because of

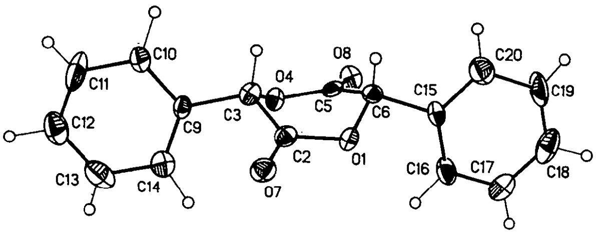

Fig. 1. View of (1) showing the atomic labeling scheme. Thermal ellipsoids are scaled to the 50% probability level. The phenyl rings bisect the O—C—C angles at C3 and C6 resulting in close H···H contacts between H atoms on C3 and C10 [H3···H10 2·30 (7) Å] and C6 and C20 [H6···H20 2·36 (9) Å].

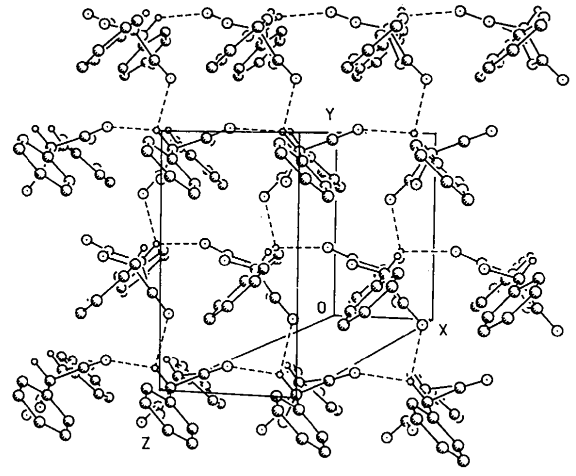

Fig. 2. View of the unit-cell packing diagram. Phenyl ring H atoms have been omitted for clarity. Dashed lines indicate C—H···O-type hydrogen bonds. The three-center hydrogen bonding is apparent from this orientation.

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* Tables of anisotropic thermal parameters, H-atom positional parameters, bond distances and angles involving the H atoms, structure factor amplitudes and a unit-cell packing diagram have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52578 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

the high melting point (531–532 K) and very low solubility of the crystalline material in organic solvents. The crystal structure of the related compound, racemic 3,6-dimethyl-1,4-dioxane-2,5-dione, has been reported (van Hummel, Harkema, Kohn & Feijen, 1982) and it also has close C—H···O contacts [H···O 2·33 (2) Å]. The occurrence and the geometry of C—H···*X*-type H bonds, where *X* is O, N or Cl, has been discussed by Taylor & Kennard (1982).

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Structure of a Dimer Ketone Formed *via* Iron Carbonyl-Promoted Coupling of 7-Phenylnorbornadiene with Carbon Monoxide

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Abstract. *trans-exo*-10,11-Diphenyl-1,4,4a,4b,5,8,8a,9a-octahydro-1,4:5,8-dimethanofluoren-9-one, $C_{27}H_{24}O$, $M_r = 364{\cdot}49$, triclinic, $P\bar{1}$, $a = 9{\cdot}673$ (2), $b = 9{\cdot}748$ (2), $c = 12{\cdot}522$ (3) Å, $\alpha = 91{\cdot}80$ (2), $\beta = 107{\cdot}12$ (2), $\gamma = 118{\cdot}23$ (1)°, $V = 973{\cdot}6$ (3) Å^3, $Z = 2$, $D_x = 1{\cdot}243$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}68$ cm^{-1}, $F(000) = 388$, $T = 295$ K, $R = 0{\cdot}0642$ for 1666 reflections. The two norbornene rings are *trans*-fused to a cyclopentanone ring. The norbornene bonds adjacent to the carbonyl average 1·577 (2) Å while those attached opposite the carbonyl average 1·558 (2) Å. The sp^2 C atoms of the norbornene moieties are pyramidalized with the two double bonds exhibiting folding angles of about 10 (1)°.

Experimental. A small, colorless crystal of dimensions 0·07 × 0·15 × 0·60 mm was mounted on a Nicolet $R3m/\mu$ update of a $P2_1$ diffractometer; data collected in the ω mode ($3 \le 2\theta \le 45°$) using a variable scan rate (4 to 29·3° min^{-1}) and graphite-monochromated Mo $K\alpha$ radiation; lattice parameters from a least-squares refinement of 25 reflections ($25{\cdot}46 \le 2\theta \le 36{\cdot}40°$); no systematic absences and intensity statistics consistent with space group $P\bar{1}$; monitored reflections (021 and $2\bar{1}\bar{2}$) showed a random maximum 2% variation during data collection; $-10 \le h \le 9$; $-10 \le k \le 10$; $0 \le l \le 13$; 3540 reflections measured, 2576 independent ($R_{\rm merge} = 0{\cdot}008$), 1666 with $I \ge 3\sigma(I)$; Lorentz–polarization corrections, ψ-scan-based empirical absorption correction (transmission factors 0·895 to 0·956); structure solved by direct methods, H atoms located in difference maps and refined with isotropic thermal parameters; final $R = 0{\cdot}0642$, $wR = 0{\cdot}0551$ for 349 parameters ($R = 0{\cdot}0885$, $wR = 0{\cdot}0673$ for all 2576 observed reflections), $S = 1{\cdot}298$, $(\Delta/\sigma)_{\max} = 0{\cdot}021$, largest peaks in the final difference map of $-0{\cdot}27$

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