The O···O spacings range from 2·67 (1) to 2·97 (1) Å and the N(1)···O*W* (1) and N(2)···O(3′) spacings have the respective values of 2·70 (1) and 2·80 (1) Å (see Table 3).

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Structure of a Photodimer of 3-Acetoxy-2-inden-1-one: 9,10-Dioxoindano[2′,3′:4,3]cyclobuta[1,2-*b*]indan-4b,4c-diyl Diacetate

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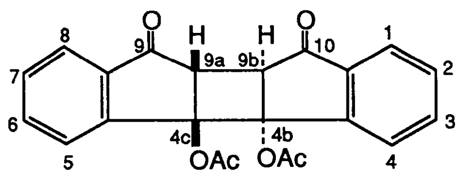
Abstract. $C_{22}H_{16}O_6$, $M_r = 376{\cdot}37$, monoclinic, $P2_1/c$, $a = 9{\cdot}555$ (3), $b = 15{\cdot}664$ (2), $c = 12{\cdot}300$ (4) Å, $\beta = 100{\cdot}08$ (2)°, $V = 1812{\cdot}5$ (5) Å^3, $Z = 4$, $D_x = 1{\cdot}379$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 8{\cdot}0$ cm^{-1}, $F(000) = 784$, room temperature, $R = 0{\cdot}047$, $wR = 0{\cdot}064$ for 3203 observed reflections [$I > 3\sigma(I)$]. The molecule exists as the *syn-trans* isomer in the crystal. The crystal structure exhibits a number of C—H···O intermolecular contacts.

Introduction. In connection with the synthesis of radermachol (Joshi, Gawad, Pelletier, Kartha & Bhandary, 1984), we carried out a photochemical reaction between 3-acetoxy-2-inden-1-one and 1,1,4,4-tetramethoxy-1,4-dihydronaphthalene. The photochemical reaction of enones with olefins has been investigated by several workers and fairly good yields of the photoaddition product have been obtained (Bryce-Smith & Gilbert, 1964; Barltrop & Hesp, 1967; Pappas & Portnoy, 1970; Maruyama, Otsuki & Naruta, 1973; Otsuki, 1976). However, in the present case, the indenone being highly reactive, rapidly dimerized to give a compound $C_{22}H_{16}O_6$ (1); the same compound was also obtained by photo reaction of the indenone. Four isomeric structures of the truxenone derivatives (*syn-cis, syn-trans, anti-cis* and *anti-trans*) are possible for the dimer. It was difficult to make a choice among the four, based only on the ^{1}H, ^{13}C and mass spectral evidence. The correct structure was therefore determined by X-ray diffraction and is reported here.



(1)

Experimental. 3-Acetoxy-2-inden-1-one was prepared by following a reported procedure (Sraga & Hunciar, 1986). The dimer was obtained by irradiating a solution of the indenone (100 mg) in benzene (400 ml) at 285 K for two hours under nitrogen with a medium pressure mercury lamp. The solvent was removed and the product crystallized from benzene to afford the dimer (63 mg). This was recrystallized

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from CH_2Cl_2:hexane to yield colorless crystals, m.p. 563–564 K. ^{1}H NMR (250 MHz, $CDCl_3$): δ 1·83 (6H, *s*), 3·20 (2H, *s*), 7·61 (2H, *t*, $J = 7·3$ Hz), 7·77 (2H, *t*, $J = 7·5$ Hz), 7·89 (2H, *d*, $J = 7·5$ Hz), 7·92 (2H, *d*, $J = 7·5$ Hz). DEPT ^{13}C NMR (62·9 MHz, $CDCl_3$): 21·2 (*q*), 51·4 (*d*), 83·5 (*s*), 124·9 (*d*), 127·7 (*d*), 130·9 (*d*), 135·1 (*d*), 137·8 (*s*), 148·0 (*s*), 169·1 (*s*), 199·0 (*s*). Three-dimensional intensity data were collected on a CAD-4 diffractometer using a crystal of dimensions 0·2 × 0·3 × 0·5 mm; the lattice parameters were refined using 25 centered reflections in the range $22 < \theta < 29°$; a total of 3915 reflections were measured of which 3615 were unique; $R_{sym} = 0·025$; reflections were measured ($2\theta = 150°$) using $\omega/2\theta$ scan; ω-scan width was calculated using the expression $(0·6 + 0·14\tan\theta)°$; the horizontal-aperture width ranged from 2·4 to 3·5 mm and the vertical-aperture width was set at 4·0 mm; range of *hkl*: *h* 0 to 11, *k* 0 to 19 and *l* −15 to 15; 3203 reflections were significant [$I > 3\sigma(I)$]; three reflections were measured every hour of X-ray exposure and showed no significant variation in their intensities during the course of data collection; no decay correction was applied; Lorentz and polarization and anisotropy of absorption using ψ-scan

Table 1. *Fractional atomic coordinates with equivalent isotropic thermal parameters for non-*H *atoms and isotropic thermal parameters for* H *atoms*

The e.s.d.'s are given in parentheses. The equivalent isotropic thermal parameter, U_{eq}, is defined as $U_{eq} = \frac{1}{3}[U_{22} + 1/\sin^2\beta(U_{11} + U_{33} + 2U_{23}\cos\beta)]$.

	x	*y*	*z*	U_{eq}/B^*(Å^2)
O1	0·7807 (1)	0·14297 (6)	0·14931 (7)	0·040 (2)
C2	0·7779 (1)	0·17490 (8)	0·2583 (1)	0·035 (3)
C3	0·7771 (1)	0·11574 (8)	0·3594 (1)	0·037 (3)
C4	0·6371 (2)	0·13233 (9)	0·3981 (1)	0·040 (3)
O4	0·5888 (1)	0·09041 (8)	0·46575 (9)	0·055 (3)
C5	0·4527 (2)	0·2542 (1)	0·3543 (2)	0·053 (4)
C6	0·4115 (2)	0·3226 (1)	0·2856 (2)	0·063 (4)
C7	0·4895 (2)	0·3470 (1)	0·2053 (2)	0·060 (4)
C8	0·6120 (2)	0·3033 (1)	0·1918 (1)	0·049 (3)
C9	0·6533 (1)	0·23405 (9)	0·2606 (1)	0·037 (3)
C10	0·5743 (2)	0·20958 (9)	0·3404 (1)	0·041 (3)
C11	0·6767 (2)	0·08861 (9)	0·1046 (1)	0·044 (3)
O11	0·5910 (1)	0·06092 (8)	0·1560 (1)	0·065 (3)
C12	0·6867 (2)	0·0689 (1)	−0·0123 (1)	0·061 (4)
O1′	0·9307 (1)	0·30005 (6)	0·29515 (8)	0·039 (2)
C2′	0·9237 (1)	0·20998 (8)	0·3126 (1)	0·034 (3)
C3′	0·9030 (1)	0·17114 (9)	0·4248 (1)	0·037 (3)
C4′	1·0350 (2)	0·11861 (9)	0·4663 (1)	0·041 (3)
O4′	1·0678 (1)	0·08941 (9)	0·55822 (9)	0·062 (3)
C5′	1·2330 (2)	0·0616 (1)	0·3665 (1)	0·047 (3)
C6′	1·2904 (2)	0·0667 (1)	0·2713 (1)	0·049 (3)
C7′	1·2304 (2)	0·1196 (1)	0·1848 (1)	0·047 (3)
C8′	1·1102 (2)	0·16785 (9)	0·1906 (1)	0·041 (3)
C9′	1·0511 (1)	0·16213 (8)	0·2859 (1)	0·034 (3)
C10′	1·1132 (2)	0·11012 (9)	0·3730 (1)	0·038 (3)
C11′	1·0365 (1)	0·34385 (9)	0·3582 (1)	0·039 (3)
O11′	1·1194 (1)	0·31142 (7)	0·43115 (9)	0·054 (3)
C12′	1·0349 (2)	0·4351 (1)	0·3234 (2)	0·058 (4)
HC3	0·802 (2)	0·054 (1)	0·351 (1)	1·3 (3)*
HC5	0·396 (2)	0·237 (1)	0·412 (2)	3·3 (5)*
HC6	0·323 (2)	0·356 (2)	0·297 (2)	4·5 (5)*
HC7	0·452 (2)	0·388 (2)	0·156 (2)	4·7 (6)*
HC8	0·669 (2)	0·320 (1)	0·136 (1)	1·8 (3)*
HC121	0·634 (3)	0·029 (2)	−0·039 (2)	7·5 (9)*
HC122	0·781 (3)	0·064 (2)	−0·033 (2)	5·5 (7)*
HC123	0·656 (4)	0·118 (3)	−0·070 (3)	11 (1)*
HC3′	0·873 (2)	0·210 (1)	0·478 (1)	2·0 (4)*
HC5′	1·273 (2)	0·024 (1)	0·428 (2)	2·8 (4)*
HC6′	1·374 (2)	0·030 (1)	0·265 (1)	2·0 (4)*
HC7′	1·271 (2)	0·122 (1)	0·121 (2)	3·2 (4)*
HC8′	1·065 (2)	0·206 (1)	0·130 (1)	1·9 (4)*
HC12′1	0·952 (3)	0·462 (2)	0·330 (3)	7·6 (8)*
HC12′2	1·113 (5)	0·462 (3)	0·367 (3)	11 (1)*
HC12′3	1·040 (5)	0·444 (3)	0·242 (4)	15 (2)*

Table 2. *Bond lengths* (Å) *and angles* (°) *involving the non-*H *atoms, with e.s.d.'s in parentheses*

O1—C2	1·437 (2)	C11—C12	1·490 (3)
O1—C11	1·349 (2)	O1′—C2′	1·431 (2)
C2—C3	1·551 (2)	O1′—C11′	1·348 (2)
C2—C9	1·513 (2)	C2′—C3′	1·553 (2)
C2—C2′	1·536 (2)	C2′—C9′	1·513 (2)
C3—C4	1·518 (2)	C3′—C4′	1·516 (2)
C3—C3′	1·585 (2)	C4′—O4′	1·210 (2)
C4—O4	1·213 (2)	C4′—C10′	1·481 (2)
C4—C10	1·476 (2)	C5′—C6′	1·380 (3)
C5—C6	1·379 (3)	C5′—C10′	1·389 (2)
C5—C10	1·392 (2)	C6′—C7′	1·392 (2)
C6—C7	1·391 (3)	C7′—C8′	1·386 (2)
C7—C8	1·391 (3)	C8′—C9′	1·390 (2)
C8—C9	1·389 (2)	C9′—C10′	1·394 (2)
C9—C10	1·393 (2)	C11′—O11′	1·201 (2)
C11—O11	1·201 (2)	C11′—C12′	1·492 (2)

C2—O1—C11	117·9 (1)	O11—C11—C12	126·7 (2)
O1—C2—C3	122·9 (1)	C2′—O1′—C11′	117·8 (1)
O1—C2—C9	111·9 (1)	C2—C2′—O1′	110·5 (1)
O1—C2—C2′	111·5 (1)	C2—C2′—C3′	90·0 (1)
C3—C2—C9	103·7 (1)	C2—C2′—C9′	115·5 (1)
C3—C2—C2′	89·9 (1)	O1′—C2′—C3′	122·4 (1)
C9—C2—C2′	115·5 (1)	O1′—C2′—C9′	113·3 (1)
C2—C3—C4	106·1 (1)	C3′—C2′—C9′	103·4 (1)
C2—C3—C3′	88·2 (1)	C3—C3′—C2′	88·1 (1)
C4—C3—C3′	112·2 (1)	C3—C3′—C4′	112·8 (1)
C3—C4—O4	125·7 (1)	C2′—C3′—C4′	106·5 (1)
C3—C4—C10	107·2 (1)	C3′—C4′—O4′	125·8 (2)
O4—C4—C10	127·1 (1)	C3′—C4′—C10′	106·9 (1)
C6—C5—C10	118·0 (2)	O4′—C4′—C10′	127·3 (1)
C5—C6—C7	121·1 (2)	C6′—C5′—C10′	118·2 (1)
C6—C7—C8	121·1 (2)	C5′—C6′—C7′	120·7 (2)
C7—C8—C9	117·8 (2)	C6′—C7′—C8′	121·3 (2)
C2—C9—C8	127·4 (1)	C7′—C8′—C9′	118·1 (1)
C2—C9—C10	111·7 (1)	C2′—C9′—C8′	127·8 (1)
C8—C9—C10	120·8 (1)	C2′—C9′—C10′	111·9 (1)
C4—C10—C5	129·1 (2)	C8′—C9′—C10′	120·3 (1)
C4—C10—C9	109·8 (1)	C4′—C10′—C5′	128·9 (1)
C5—C10—C9	121·0 (1)	C4′—C10′—C9′	109·8 (1)
O1—C11—O11	122·3 (1)	C5′—C10′—C9′	121·3 (1)
O1—C11—C12	111·0 (1)	O1′—C11′—O11′	122·9 (1)
O1′—C11′—C12′	110·8 (1)	O11′—C11′—C12′	126·2 (1)

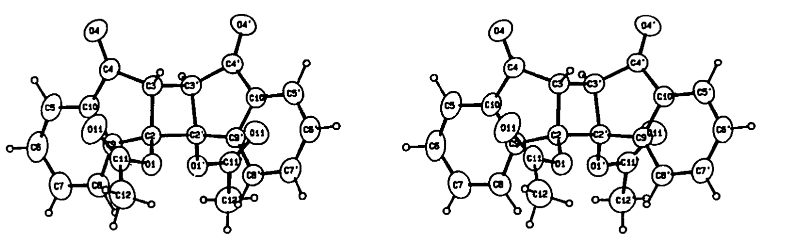

Fig. 1. A stereoview of the molecule showing the atomic numbering scheme. The thermal ellipsoids are at 50% probability level.

corrections (North, Phillips & Mathews, 1968) were applied to the data; the average, minimum and maximum transmissions are 0·92, 0·82 and 0·99, respectively.

The structure was solved by the application of direct methods using the program *SHELXS*86 (Sheldrick, 1985); the structural parameters were refined using full-matrix least-squares refinement; all H atoms were located from a difference electron density map computed at an R of 7·2%; the H atoms were then included in the refinement procedure with individual isotropic thermal parameters; the R value at the last cycle of refinement, with the non-H atoms treated anisotropically and the H-atoms isotropically, is 0·047 for 3203 observed reflections; wR is 0·064; the function minimized in the least-squares procedure was $\sum w(|F_o|^2 - 1/k|F_c|^2)$, where $w = 4|F_o|^2/\sigma^2(|F_o|^2)$; $\sigma^2(|F_o|^2) = [\sigma(I) + s^2I^2]\mathrm{Lp}$; $s = 0·02$; $\sigma(I)$ is based on intensity statistics and K is the scale factor; $(\Delta/\sigma)_{max} < 0·01$ for all atoms in the last cycle; largest features in the difference electron density map at $R = 0·047$ are 0·21 and −0·32 e Å^{-3}; $S = 2·8$; atomic scattering factors were from *International Tables for X-ray Crystallography* (1974); program for least-squares refinement, Fourier analysis and *ORTEP* (Johnson, 1976) as in *SDP* (B. A. Frenz & Associates, Inc., 1982).

Discussion. The final positional and isotropic equivalent thermal parameters for all atoms are listed in Table 1.* The bond lengths and angles are given in Table 2. The molecule is a *syn-trans* isomer as shown in Fig. 1. The molecule exhibits remarkable correspondence between the two monomeric (Table 2) units except at the cyclobutane ring {the bond length C3—C3′ [1·585 (2) Å] is significantly longer (> 20σ) compared to C2—C2′ [1·536 (2) Å]}. The molecule does not exhibit twofold symmetry that is apparent in the chemical structure. The conformation of the cyclobutane rings is defined by two parameters, δ denoting the departure of the sum of the endocyclic angles from 360°, and θ, the angle of puckering. In the present structure, the cyclobutane ring is puckered [δ 3·8 (2) and θ 20·9 (2)°; Adman & Margulis, 1969; Margulis, 1969; Shirrell & Williams, 1973]. The phenyl rings are planar with the largest deviation of any atom from the best plane being less than 0·008 Å, but the pentenone rings are not planar with deviations up to 0·1 Å (for C3 and C3′ atoms) from the best plane. The acetoxy groups are also planar and are oriented at right angles to the plane defined by the phenyl ring. Such an orientation brings the O11 and O11′ atoms in close proximity to the phenyl rings [2·648 (1) and 2·638 (1) Å, respectively]. It has been shown previously that the O atoms of ester groups have a strong tendency to overlap with an unsaturated ring such as a phenyl ring; this type of interaction has been put to good use in the photopolymerization of phenyl diacrylic acids (Nakanishi, Jones, Thomas, Hasegawa & Rees, 1980). The molecule lacks any proton donor groups such as O—H, N—H, *etc.*, but has a total of six O atoms. It is, therefore, not surprising to observe C—H···O van der Waals distances in the crystal structure (Taylor & Kennard, 1982). These are listed in Table 3. However, none of the contacts meet the criteria of short distance [$d > 0·3$ Å where $d = v(\mathrm{H}) + v(\mathrm{O}) - r(\mathrm{H}\cdots\mathrm{O})$; $r(\mathrm{H}\cdots\mathrm{O})$, $v(\mathrm{H})$ and $v(\mathrm{O})$ are the distances between the interacting atoms and the respective van der Waals radii for H and O atoms] to be classified as a hydrogen bond. The packing of the molecules in the unit cell is shown in Fig. 2. To our knowledge, no work has been carried out earlier on the X-ray crystal structure determination of truxenones.

Table 3. C—H···O *intermolecular distances* (< 3 Å) *and angles* (°) *observed in the crystal structure*

D	H	O	*D*—H	H···O	*D*···O	*D*—H···O	Code*
C5′	HC5′	O4	1·00 (2)	2·45 (2)	3·405 (2)	161 (2)	(III) (201)
C8′	HC8′	O11′	1·00 (2)	2·61 (2)	3·224 (2)	120 (2)	(IV) (00-1)
C12′	HC12′3	O4′	1·00 (5)	2·41 (5)	3·354 (2)	157 (4)	(IV) (00-1)
C8	HC8	O4	0·98 (2)	2·52 (2)	3·215 (2)	128 (2)	(IV) (00-1)
C3	HC3	O4′	0·99 (2)	2·71 (2)	3·609 (2)	149 (2)	(III) (201)
C6′	HC6′	O11	1·00 (2)	2·70 (2)	3·419 (2)	129 (2)	(I) (100)
C7′	HC7′	O11′	0·95 (2)	2·73 (2)	3·297 (2)	119 (2)	(IV) (00-1)
C12	HC121	O11	0·85 (3)	2·74 (3)	3·558 (2)	163 (2)	(III) (100)

* (I) x, y, z; (III) $-x, -y, -z$; (IV) $x, \frac{1}{2}-y, \frac{1}{2}+z$.

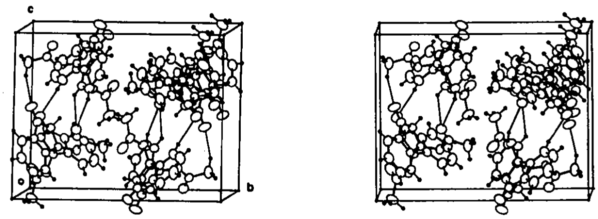

Fig. 2. Packing of the molecules in the unit cell. The shortest H···O distances are shown as thin lines.

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* Lists of bond lengths and angles involving H atoms, structure factors, anisotropic thermal parameters and results of least-squares-plane calculations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52836 (33 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Tetramethyl Reductic Acid (TMRA, 4,4,5,5-Tetramethyl-2,3-dihydroxy-2-cyclopenten-1-one), a Reductate Ligand Involved in Dioxygen Activation by Multinuclear Iron Complexes

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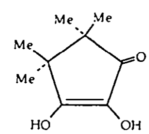
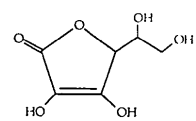
Abstract. $C_9H_{14}O_3$, $M_r = 170·21$, orthorhombic, *Pbca*, $a = 11·651$ (2), $b = 12·279$ (2), $c = 26·257$ (4) Å, $V = 3756$ Å^3, $Z = 16$, $D_x = 1·204$, $D_m = 1·19$ (1) g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 7·01$ cm^{-1}, $F(000) = 1472$, $T = 297$ (1) K, $R = 0·051$ for 2152 observed reflections with $F > 6\sigma(F)$. The title compound adopts a well-defined conjugated structure corresponding to one of its expected resonance forms. There are two independent molecules in the asymmetric unit owing to two different intermolecular hydrogen-bonding modes.

Introduction. Tetramethyl reductic acid (TMRA) was synthesized several years ago (Hesse & Wehling, 1964) and later used in a photographic developer (Bloom & Cramer, 1971). As an analog of ascorbic acid (AA), its functions as both reductant and ligand in many transition-metal-ion-mediated chemical and biological redox processes have been of much current interest (Bryan, Pell, Kumar, Clarke, Rodriguez, Sherban & Charkoudian, 1988). Work in our laboratory has indicated that complexes of TMRA with several diiron(III) salts can catalyze oxidation of saturated hydrocarbons by molecular oxygen (Roth, 1988). Although spectroscopic and electrochemical properties of TMRA have been studied (Hesse & Wehling, 1964; Inbar, Ehret & Norland, 1987), a definitive structural characterization has not been reported to date. Here, we present the crystal and molecular structure.



TMRA



AA

Experimental. Tetramethyl reductic acid was obtained from Polaroid Corporation. Crystals were grown from acetonitrile at 253 K. One colorless, transparent needle of approximate dimensions 0·3 × 0·35 × 0·5 mm was mounted in a capillary tube. X-ray data were collected on an Enraf–Nonius CAD-4 diffractometer with Ni-filtered Cu $K\alpha$ radiation. The cell parameters were measured from a least-squares fit of 24 high-angle reflections ($2\theta > 55°$). Intensity data were collected by $\omega/2\theta$ scans using a peak scan width of $(0·80 + 0·15\tan\theta)°$ with an additional 25% before and after each reflection for background determination. The scan time was 10–150 s. A total of 4655 reflections in the 2θ range 2–130° (h 0–13, k 0–14, l −30–30) were measured. Three standard reflections monitored every 3600 s of exposure time showed no significant variation. No correction was made for extinction or absorption. 2152 unique reflections observed with $F > 6\sigma(F)$, R_{int}

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