Refinement

Refinement on F
$R = 0.0384$
$wR = 0.0366$
$S = 2.339$
1552 reflections
146 parameters
H atoms riding, C—H 0.98 Å
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 0.16$ e Å^{-3}
$\Delta\rho_{min} = -0.10$ e Å^{-3}
Extinction correction: Zachariasen (1968) type 2 Gaussian isotropic
Extinction coefficient: $8.4\,(3) \times 10^{-6}$
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

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

	x	y	z	U_{eq}
O(1)	−0.0317 (1)	0.0794 (1)	−0.2880 (1)	0.0919 (5)
C(1)	0.0528 (2)	0.0028 (2)	−0.2656 (2)	0.0608 (6)
C(2)	0.1467 (2)	0.0050 (2)	−0.1551 (2)	0.0635 (6)
C(3)	0.1278 (2)	0.1068 (2)	−0.0752 (2)	0.0597 (6)
C(4)	0.2286 (2)	0.1066 (2)	0.0318 (2)	0.0609 (6)
C(5)	0.2105 (2)	0.2100 (2)	0.1117 (2)	0.0691 (6)
C(6)	0.3184 (2)	0.2166 (2)	0.2136 (2)	0.0582 (6)
C(7)	0.4108 (2)	0.3087 (2)	0.2258 (2)	0.0696 (7)
C(8)	0.5120 (2)	0.3136 (2)	0.3173 (2)	0.0737 (7)
C(9)	0.5256 (2)	0.2265 (2)	0.3998 (2)	0.0602 (6)
C(10)	0.4317 (2)	0.1353 (2)	0.3890 (2)	0.0642 (6)
C(11)	0.3296 (2)	0.1291 (2)	0.2974 (2)	0.0646 (6)
C(12)	0.6403 (2)	0.2283 (2)	0.4975 (2)	0.0762 (7)
C(13)	0.7794 (2)	0.2177 (2)	0.4586 (2)	0.0762 (7)
C(14)	0.7974 (2)	0.1067 (2)	0.3905 (2)	0.0732 (7)
C(15)	0.9334 (2)	0.1000 (2)	0.3445 (2)	0.0728 (7)

Table 2. *Selected bond lengths* (Å) *and angles* (°)

C—C (aliphatic)	1.495–1.516 (3)
C—C (aromatic)	1.373–1.386 (3)
C=O	1.207 (2)
C—C_{sp^3}—C	113.5–116.2 (2)
C—C_{sp^2}—C	117.1–122.0 (2)

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1990). Program(s) used to solve structure: *TEXSAN*. Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

We thank the Natural Sciences and Engineering Research Council of Canada for financial support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, least-squares-planes data and complete geometry have been deposited with the IUCr (Reference: FG1093). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1995). C**51**, 2688–2690

3,3,18,18-Tetramethyl-1,5,16,20-tetraoxadispiro[5.8.5.8]octacosane

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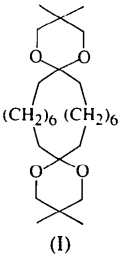
Abstract

The centrosymmetric molecule, $C_{28}H_{52}O_4$, contains an 18-membered ring, with two spiro-fused 1,3-dioxa six-membered rings.

Comment

The title compound, (I), was obtained as part of a study of the photochemistry of macrocyclic diketones (Lewis, Rettig, Scheffer, Trotter & Wireko, 1990; Lewis, Rettig, Scheffer & Trotter, 1991), in an (unsuccessful) effort to protect one of the two ketone groups by forming a mono-acetal.



(I)

The molecule lies across a crystallographic center of inversion and contains a central 18-membered carbocyclic ring, spiro-fused to two six-membered 1,3-dioxacyclohexane rings. The 18-membered ring contains ten bonds with *trans* conformation [torsion angles = 171.5–179.4 (2)°] and eight bonds with *gauche* conformation [56.2–70.0 (2)°]. The six-membered rings have chair conformations [torsion angles = 53.4–56.7 (1)°].

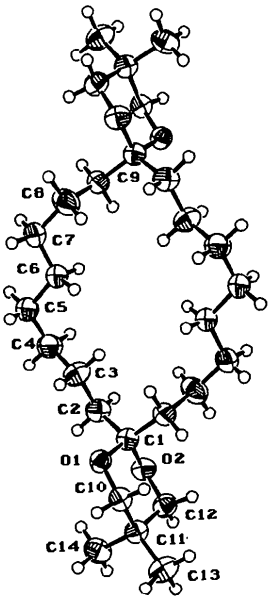


Fig. 1. View of the molecule with 33% probability displacement ellipsoids and the crystallographic numbering system.

Experimental

The title compound was prepared according to the procedures described by Lewis (1993).

Crystal data

$C_{28}H_{52}O_4$
$M_r = 452.72$
Triclinic
$P\bar{1}$
$a = 10.718\,(1)$ Å
$b = 13.050\,(1)$ Å
$c = 5.5074\,(4)$ Å
$\alpha = 90.27\,(1)°$
$\beta = 104.32\,(1)°$
$\gamma = 109.86\,(1)°$
$V = 698.6\,(1)$ Å^3
$Z = 1$
$D_x = 1.076$ Mg m^{-3}
Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 25 reflections
$\theta = 46.4$–$57.4°$
$\mu = 0.51$ mm^{-1}
$T = 294$ K
Needle
0.40 × 0.25 × 0.25 mm
Colorless

Data collection

AFC-6S diffractometer
ω–2θ scans
Absorption correction: refined from ΔF (*DIFABS*; Walker & Stuart, 1983)
$T_{min} = 0.72$, $T_{max} = 1.00$
3165 measured reflections
2844 independent reflections
2325 observed reflections [$I > 3\sigma(I)$]
$R_{int} = 0.019$
$\theta_{max} = 77.5°$
$h = 0 \rightarrow 13$
$k = -15 \rightarrow 15$
$l = -6 \rightarrow 6$
3 standard reflections monitored every 250 reflections
intensity decay: none

Refinement

Refinement on F
$R = 0.042$
$wR = 0.056$
$S = 2.9$
2325 reflections
250 parameters
All H-atom parameters refined
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.17$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}
Extinction correction: Zachariasen (1968) type 2 Gaussian isotropic
Extinction coefficient: 1.4×10^{-4}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

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

	x	y	z	U_{eq}
O1	0.40413 (8)	0.35243 (7)	0.3836 (2)	0.0464 (4)
O2	0.2246 (1)	0.26671 (7)	0.0250 (2)	0.0521 (4)
C1	0.3291 (1)	0.2527 (1)	0.2264 (2)	0.0450 (5)
C2	0.4303 (2)	0.2328 (1)	0.0986 (3)	0.0552 (6)
C3	0.5512 (2)	0.2123 (2)	0.2722 (3)	0.0633 (8)
C4	0.6460 (2)	0.1885 (2)	0.1336 (4)	0.0709 (8)
C5	0.7689 (2)	0.1689 (1)	0.3052 (5)	0.0743 (9)
C6	0.7318 (2)	0.0712 (1)	0.4559 (4)	0.0619 (6)
C7	0.8525 (2)	0.0424 (1)	0.6016 (5)	0.0738 (8)
C8	0.8132 (2)	−0.0502 (1)	0.7676 (4)	0.0726 (8)
C9	0.7303 (1)	−0.1608 (1)	0.6187 (3)	0.0504 (5)
C10	0.3216 (1)	0.4001 (1)	0.4786 (2)	0.0469 (5)
C11	0.2141 (1)	0.4201 (1)	0.2648 (2)	0.0493 (5)
C12	0.1353 (1)	0.3110 (1)	0.1048 (3)	0.0550 (6)
C13	0.1157 (2)	0.4561 (2)	0.3713 (4)	0.0713 (9)
C14	0.2851 (2)	0.5069 (1)	0.1107 (3)	0.0631 (6)

Table 2. *Selected bond lengths* (Å) *and angles* (°)

C—C	1.510–1.527 (2)
C—O	1.423–1.430 (2)
C—C—C/O	104.6–115.4 (1)
C—O—C	114.1, 114.8 (1)

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1990). Program(s) used to solve structure: *TEXSAN*. Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

We thank the Natural Sciences and Engineering Research Council of Canada for financial support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1091). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Lewis, T. J., Rettig, S. J., Scheffer, J. R., Trotter, J. & Wireko, F. C. (1990). *J. Am. Chem. Soc.* **112**, 3679–3680.

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Molecular Structure Corporation (1990). *TEXSAN. Single Crystal Structure Analysis Package*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
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Acta Cryst. (1995). C**51**, 2690–2691

Dimethyl 1-Chloro-4b,8b,8c,8d-tetrahydrodibenzo[*a*,*f*]cyclopropa[*cd*]pentalene-8c,8d-dicarboxylate

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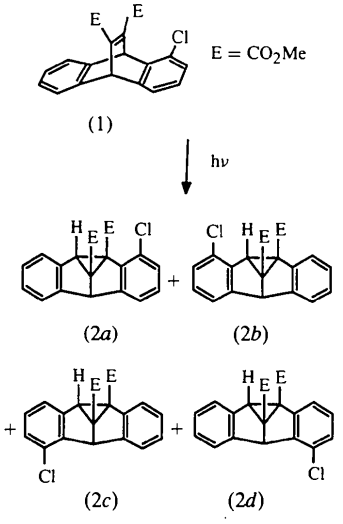
(*Received* 13 *April* 1995; *accepted* 20 *June* 1995)

Abstract

The title compound, $C_{20}H_{15}ClO_4$, is one of the photoproducts of the photolysis of a 1-chlorodibenzobarrelene diester. The geometry and dimensions of the title molecule are similar to those of related materials.

Comment

The 1-chlorodibenzobarrelene diester (1) undergoes a di-π-methane reaction in solution and in the solid state; four regioisomeric dibenzosemibullvalene photoproducts [(2*a*)–(2*d*)] are possible. The products are difficult to separate by column chromatography, with broad overlapping GC signals. Only one product could be isolated and its molecular structure was determined by X-ray analysis to be that of (2*a*). The details of the photochemical pathways have not been established.



The mechanism for the formation of (2*a*) from (1) involves formation of a new C11—C9a bond (dibenzobarrelene numbering system), the breaking of bond C9—C9a and finally formation of a C9—C12 bond. The molecular structure of (2*a*) (Fig. 1 and Table 2) is similar to that of related materials (Garcia-Garibay, Scheffer, Trotter & Wireko, 1990; Pokkuluri, Scheffer & Trotter, 1993). The external angles at the ring junctions are large [128.4–130.4 (1)°] as a result of the additional steric strain due to the formation of the three-membered ring, with the largest angle adjacent to the Cl substituent.

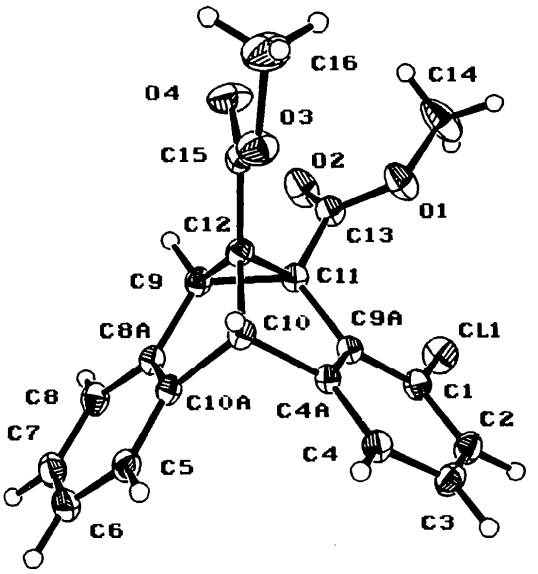


Fig. 1. View of the molecule with 33% probability displacement ellipsoids; the dibenzobarrelene numbering system is used.

Experimental

The title compound was prepared according to the procedures described by Yang (1993).

Crystal data

$C_{20}H_{15}ClO_4$
M_r = 354.79
Triclinic
$P\bar{1}$
a = 9.756 (1) Å
b = 10.556 (1) Å
c = 8.830 (1) Å
α = 107.22 (1)°
β = 105.31 (1)°
γ = 95.40 (1)°
V = 823.0 (1) Å^3
Z = 2
D_x = 1.432 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.54178 Å
Cell parameters from 25 reflections
θ = 48.2–55.5°
μ = 2.3 mm^{-1}
T = 294 K
Prism
0.40 × 0.35 × 0.25 mm
Colorless

Data collection

AFC-6*S* diffractometer
ω–2θ scans
R_{int} = 0.023
θ_{max} = 77.5°