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Tricyclo[22.2.2.2^{11,14}]triaconta-11,13,24,26,27,29-hexaene-6,19-dione

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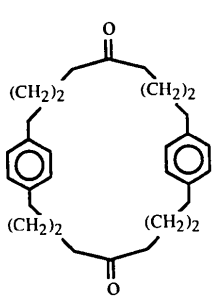
Abstract

The title molecule, $C_{30}H_{40}O_2$, contains a centrosymmetric 26-membered ring and has a conformation in which O···γ-H distances are in the range 4.6–5.0 Å, too long for H-atom abstraction, and in accord with the lack of photochemical reactivity in the solid state.

Comment

The title compound, (I), was examined as part of a study of the photochemistry of macrocyclic diketones (Lewis, Rettig, Scheffer, Trotter & Wireko, 1990; Lewis, Rettig, Scheffer & Trotter, 1991). Photolysis in solution gives three Norrish type II photoproducts (cyclization and cleavage), but the compound is not photochemically reactive in the solid state.



(I)

The molecule crystallizes with a conformation in which the O···γ-H distances are in the range 4.6–5.0 Å [O···H on C(4) and C(13) in Fig. 1], much too long for H-atom abstraction. Other conformations presumably give rise to the reactivity in solution (Lewis *et al.*, 1990).

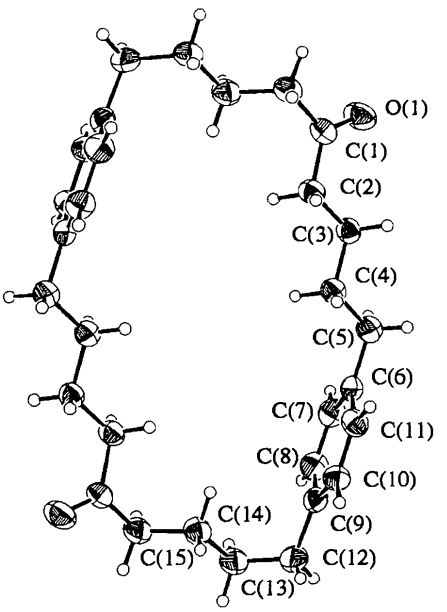


Fig. 1. View of the title molecule showing the crystallographic numbering system. Displacement ellipsoids are drawn at the 33% probability level.

Experimental

Crystals of the title compound were obtained according to the procedure described by Yang (1993).

Crystal data

$C_{30}H_{40}O_2$
M_r = 432.64
Monoclinic
$P2_1/c$
a = 9.920 (1) Å
b = 11.209 (1) Å
c = 11.755 (1) Å
β = 96.93 (1)°
V = 1297.5 (1) Å^3
Z = 2
D_x = 1.107 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.54178 Å
Cell parameters from 25 reflections
θ = 34.1–44.1°
μ = 0.48 mm^{-1}
T = 294.2 K
Prism
0.45 × 0.25 × 0.20 mm
Colorless

Data collection

AFC-6*S* diffractometer
ω–2θ scans
Absorption correction:
ψ scans (*TEXSAN*; Molecular Structure Corporation, 1990)
T_{min} = 0.92, T_{max} = 1.00
2945 measured reflections
2640 independent reflections
1552 observed reflections
[$I > 3\sigma(I)$]
R_{int} = 0.039
θ_{max} = 77.5°
h = 0 → 12
k = 0 → 13
l = −14 → 14
3 standard reflections
monitored every 250 reflections
intensity decay: none

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*.

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

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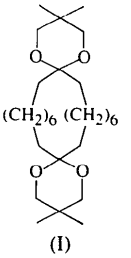
Abstract

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)°].