Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and torsion angles, together with stereo molecular and packing diagrams have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71600 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CD1061]

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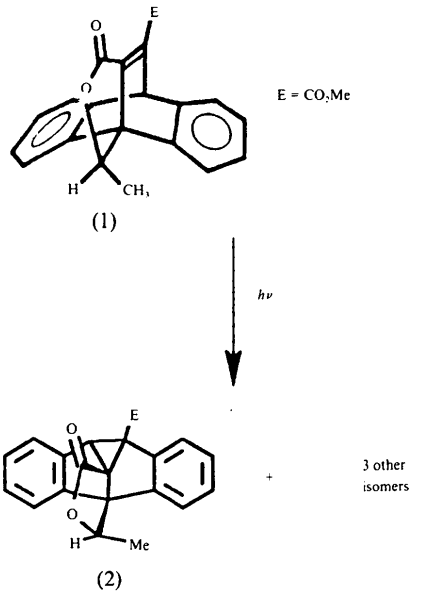

has now been determined by X-ray methods. The general geometry and dimensions of molecule (2) are similar to those of related materials (Pokkuluri, Scheffer & Trotter, 1993, 1994).

Acta Cryst. (1994). C**50**, 281–283

Structure of a Cyclopropapentalene Photolysis Product of a Dibenzobarrelene Ester Lactone

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Abstract

Photolysis of the dibenzobarrelene ester lactone, $C_{21}H_{16}O_4$ (1), can produce several products, the structure of one of which, 8b-methyl 4b-(1-hydroxyethyl)-4b,8b,8c,8d-tetrahydrodibenzo[*a*,*f*]cyclopropa[*cd*]pentalene-8b,8c-dicarboxylate carbolactone (2), has been determined by X-ray methods. The molecule of (2) contains a three-membered ring and has geometry and dimensions similar to those of related materials.

Comment

Photolysis of (1) can give four photoproducts, three of which have been isolated (Chen, Pokkuluri, Scheffer & Trotter, 1992). The structure of one of the products, (2),

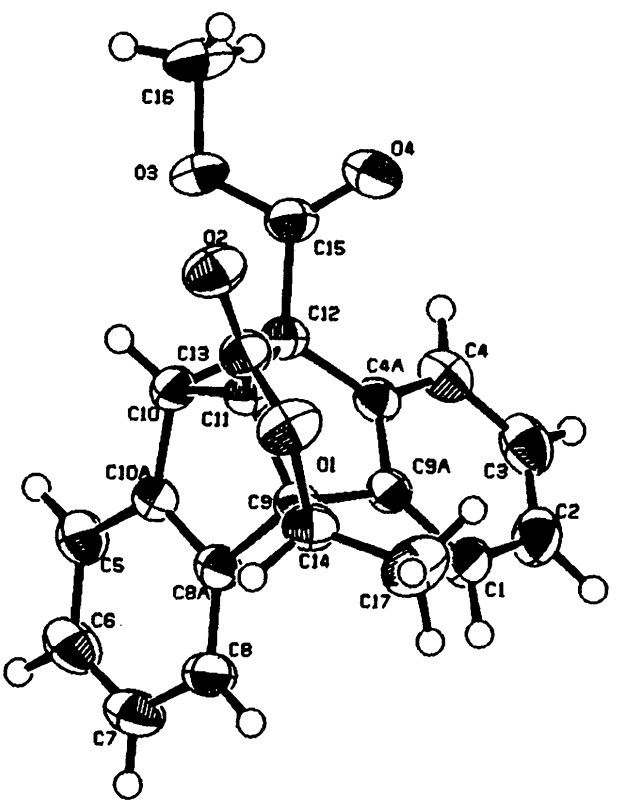

Fig. 1. View of the molecule with 50% probability ellipsoids.

Experimental

Crystal data

$C_{21}H_{16}O_4$
M_r = 332.35
Monoclinic
$P2_1/n$
a = 8.1129 (9) Å
b = 13.1445 (9) Å
c = 15.646 (1) Å
β = 98.162 (8)°
V = 1651.6 (2) Å^3
Z = 4
D_x = 1.34 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 25 reflections
θ = 41-48°
μ = 0.71 mm^{-1}
T = 294 K
Prism
0.35 × 0.30 × 0.25 mm
Colourless
Crystal source: Chen (1991)

Data collection

Rigaku AFC-6 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
(T_{min} = 0.97, T_{max} = 1.00)
3448 measured reflections
3332 independent reflections
2389 observed reflections [$I > 3\sigma(I)$]
R_{int} = 0.027
θ_{max} = 77.5°
$h = -10 \rightarrow 10$
$k = 0 \rightarrow 16$
$l = 0 \rightarrow 19$
3 standard reflections monitored every 150 reflections
intensity variation: none

Refinement

Refinement on F
R = 0.042
wR = 0.065
S = 1.96
2389 reflections
227 + 64 H parameters
H-atom parameters refined
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{max}$ = 0.006
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Extinction correction: *TEXSAN* (Molecular Structure Corporation, 1990)
Extinction coefficient: 0.54 (5) × 10^{-5}
Atomic scattering factors from *TEXSAN*

Data collection, cell refinement, data reduction, structure solution and refinement, and representation were carried out using *TEXSAN* (Molecular Structure Corporation, 1990). The structure was determined by direct methods.

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

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

	x	y	z	U_{eq}
O1	0.0576 (2)	0.0032 (1)	0.5968 (1)	0.051
O2	0.2313 (2)	0.1094 (1)	0.5427 (1)	0.056
O3	0.6542 (2)	0.1009 (1)	0.6173 (1)	0.059
O4	0.5672 (3)	0.1494 (1)	0.7394 (1)	0.071
C1	0.2799 (3)	−0.1668 (2)	0.8349 (1)	0.044
C2	0.3829 (3)	−0.1561 (2)	0.9132 (1)	0.052
C3	0.5240 (3)	−0.0979 (2)	0.9196 (2)	0.054
C4	0.5708 (3)	−0.0496 (2)	0.8485 (1)	0.046
C4*A*	0.4673 (2)	−0.0591 (1)	0.7700 (1)	0.035
C5	0.5672 (3)	−0.2724 (2)	0.5825 (2)	0.049
C6	0.5116 (4)	−0.3722 (2)	0.5850 (2)	0.055
C7	0.3632 (3)	−0.3950 (2)	0.6140 (2)	0.051
C8	0.2651 (3)	−0.3192 (2)	0.6423 (1)	0.044
C8*A*	0.3175 (2)	−0.2192 (1)	0.6379 (1)	0.036
C9	0.2432 (2)	−0.1219 (1)	0.6688 (1)	0.033
C9*A*	0.3206 (2)	−0.1150 (1)	0.7636 (1)	0.034
C10	0.4910 (3)	−0.0841 (2)	0.6039 (1)	0.037
C10*A*	0.4675 (2)	−0.1958 (2)	0.6083 (1)	0.037
C11	0.3331 (2)	−0.0387 (1)	0.6255 (1)	0.033
C12	0.4924 (2)	−0.0151 (1)	0.6849 (1)	0.035
C13	0.2104 (3)	0.0335 (2)	0.5824 (1)	0.041
C14	0.0580 (3)	−0.0978 (2)	0.6382 (1)	0.041
C15	0.5730 (3)	0.0871 (2)	0.6841 (1)	0.042
C16	0.7434 (5)	0.1963 (3)	0.6143 (3)	0.076
C17	−0.0567 (3)	−0.0931 (3)	0.7047 (2)	0.060

Table 2. *Selected geometric parameters* (Å, °)

C1—C2	1.389 (3)	C9—C11	1.526 (3)
C1—C9*A*	1.386 (3)	C9—C14	1.544 (3)
C2—C3	1.369 (4)	C10—C10*A*	1.483 (3)
C3—C4	1.380 (4)	C10—C11	1.495 (3)
C4—C4*A*	1.392 (3)	C10—C12	1.557 (3)
C4*A*—C9*A*	1.390 (3)	C11—C12	1.513 (3)
C4*A*—C12	1.492 (3)	C11—C13	1.468 (3)
C5—C6	1.390 (3)	C12—C15	1.495 (3)
C5—C10*A*	1.387 (3)	C14—C17	1.492 (3)
C6—C7	1.379 (4)	C13—O1	1.351 (3)
C7—C8	1.386 (4)	C13—O2	1.200 (3)
C8—C8*A*	1.386 (3)	C14—O1	1.477 (3)
C8*A*—C9	1.522 (3)	C15—O3	1.325 (3)
C8*A*—C10*A*	1.396 (3)	C15—O4	1.197 (3)
C9—C9*A*	1.529 (3)	C16—O3	1.452 (3)
C2—C1—C9*A*	118.6 (2)	C5—C10*A*—C8*A*	120.6 (2)
C1—C2—C3	120.9 (2)	C5—C10*A*—C10	128.4 (2)
C2—C3—C4	121.4 (2)	C8*A*—C10*A*—C10	110.9 (2)
C3—C4—C4*A*	118.0 (2)	C9—C11—C10	107.1 (2)
C4—C4*A*—C9*A*	120.9 (2)	C9—C11—C12	107.1 (1)
C4—C4*A*—C12	127.7 (2)	C9—C11—C13	109.4 (2)
C9*A*—C4*A*—C12	111.4 (2)	C10—C11—C12	62.4 (1)
C6—C5—C10*A*	118.2 (2)	C10—C11—C13	134.5 (2)
C5—C6—C7	121.1 (2)	C12—C11—C13	127.9 (2)
C6—C7—C8	121.1 (2)	C4*A*—C12—C10	121.0 (2)
C7—C8—C8*A*	118.1 (2)	C4*A*—C12—C11	104.9 (2)
C8—C8*A*—C9	130.0 (2)	C4*A*—C12—C15	118.1 (2)
C8—C8*A*—C10*A*	120.9 (2)	C10—C12—C11	58.2 (1)
C9—C8*A*—C10*A*	108.9 (2)	C10—C12—C15	118.0 (2)
C8*A*—C9—C9*A*	103.4 (2)	C11—C12—C15	121.0 (2)
C8*A*—C9—C11	103.0 (2)	C11—C13—O1	108.1 (2)
C8*A*—C9—C14	119.1 (2)	C11—C13—O2	129.7 (2)
C9*A*—C9—C11	103.4 (1)	O1—C13—O2	122.1 (2)
C9*A*—C9—C14	122.4 (2)	C9—C14—C17	117.9 (2)
C11—C9—C14	102.8 (2)	C9—C14—O1	105.2 (2)
C1—C9*A*—C4*A*	120.1 (2)	C17—C14—O1	107.8 (2)
C1—C9*A*—C9	129.8 (2)	C12—C15—O3	113.4 (2)
C4*A*—C9*A*—C9	109.3 (2)	C12—C15—O4	123.1 (2)
C10*A*—C10—C11	105.4 (2)	O3—C15—O4	123.5 (2)
C10*A*—C10—C12	121.7 (2)	C13—O1—C14	113.2 (2)
C11—C10—C12	59.4 (1)	C15—O3—C16	116.7 (2)

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

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and stereo diagrams have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71585 (32 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BR1048]

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Acta Cryst. (1994). C**50**, 283–284

Potential β-Blockers. I. 2-*tert*-Butyl-1,2,3,4-tetrahydro-4,6,8-trihydroxyisoquinolinium Sulfate Dihydrate

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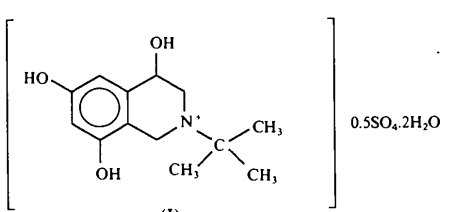
Abstract

The title molecule, $C_{13}H_{20}NO_3^+.0.5SO_4^{2-}.2H_2O$, consists of two six-membered condensed rings with the *tert*-butyl group in position 2. The aliphatic ring is in an envelope conformation. The five-atom planar part of the ring and the plane of the aromatic ring make an angle of 171.5 (1)° with respect to each other. The *tert*-butyl group is equatorial in relation to the ring. The sulfate ion lies on the twofold axis. The structure contains a three dimensional net of hydrogen bonds.

Comment

This paper commences reports on the structures of new derivatives of 1,2,3,4-tetrahydroisoquinoline-4,6,8-triol as potential β-blockers. The title compound (I) was synthesized in the Institute of Chemistry and Technology of Drugs, University of Medicine at Łódź by the simple Pictet–Spengler reaction of terbutaline, a well known β-adrenergic agonist, with formaldehyde (Brzezińska, 1994).



(I)

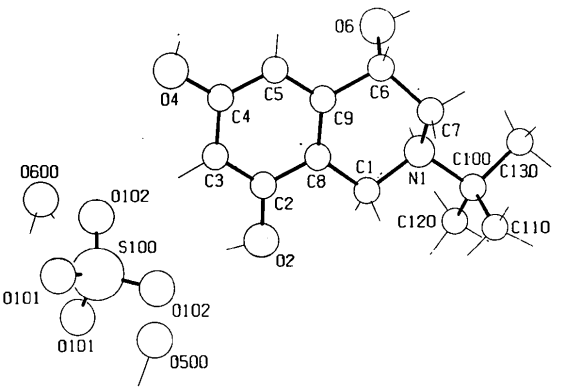


Fig. 1. View of the title compound with the atom-numbering scheme.

Experimental

Crystal data

$C_{13}H_{20}NO_3^+.0.5SO_4^{2-}.2H_2O$
M_r = 322.36
Orthorhombic
Pnca
a = 15.570 (3) Å
b = 16.579 (1) Å
c = 11.897 (1) Å
V = 3071.0 (7) Å^3
Z = 8
D_x = 1.3901 (3) Mg m^{-3}
D_m = 1.428 Mg m^{-3}
D_m measured by flotation
Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 25 reflections
θ = 15–35°
μ = 1.508 mm^{-1}
Room temperature
Thick tabular
0.3 × 0.2 × 0.1 mm
Colourless
Crystal source: slow evaporation of water at room temperature

Data collection

Kuma K4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
5706 measured reflections
2810 independent reflections
1871 observed reflections [$I > 3\sigma(I)$]
R_{int} = 0.028
θ_{max} = 80°
h = 0 → 19
k = −20 → 0
l = −15 → 13
2 standard reflections
frequency: 100 min
intensity variation: none

Refinement

Refinement on F
R = 0.0434
wR = 0.042
S = 1.08
1871 reflections
281 parameters
Unit weights applied
$(\Delta/\sigma)_{max}$ = 0.079
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}
Atomic scattering factors from *CRULER* (Rizzoli, Sangermano, Calestani & Andreetti, 1986)

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

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

	x	y	z	U_{eq}
S100	3/4	1/2	0.0701 (1)	0.0459 (3)
O101	0.6917 (1)	0.5463 (1)	−0.0014 (2)	0.0669 (8)
O102	0.7000 (2)	0.4453 (1)	0.1406 (2)	0.0744 (8)