Table 3. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *for* (II)

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

	x	y	z	$U_{\rm eq}$
S1	0.3790 (2)	1.1861 (1)	0.95189 (4)	0.0640 (4)
S2	0.4926 (2)	0.92319 (9)	0.93016 (3)	0.0446 (3)
S3	0.3916 (2)	0.80382 (9)	0.88998 (3)	0.0455 (3)
S4	0.5492 (2)	0.6850 (1)	0.81147 (4)	0.0658 (4)
P1	0.3080 (2)	1.06342 (9)	0.91801 (3)	0.0434 (3)
P2	0.6322 (2)	0.78518 (9)	0.85192 (3)	0.0439 (3)
O1	0.0808 (4)	1.0162 (2)	0.91926 (8)	0.0485 (8)
O2	0.3197 (6)	1.0791 (2)	0.87300 (7)	0.068 (1)
O3	0.8239 (5)	0.7475 (2)	0.87654 (8)	0.0492 (8)
O4	0.6932 (5)	0.9139 (2)	0.84200 (8)	0.0536 (8)
C1	−0.0081 (7)	0.9803 (3)	0.9544 (1)	0.044 (1)
C2	−0.0148 (7)	0.8650 (3)	0.9623 (1)	0.049 (1)
C3	−0.1116 (7)	0.8292 (4)	0.9954 (1)	0.060 (1)
C4	−0.2020 (8)	0.9079 (4)	1.0194 (1)	0.066 (1)
C5	−0.1944 (9)	1.0242 (4)	1.0107 (1)	0.068 (1)
C6	−0.0969 (7)	1.0615 (4)	0.9778 (1)	0.058 (1)
C7	0.3764 (7)	1.1758 (3)	0.8511 (1)	0.047 (1)
C8	0.2455 (9)	1.2054 (4)	0.8222 (1)	0.062 (1)
C9	0.297 (1)	1.2934 (4)	0.7977 (2)	0.093 (2)
C10	0.469 (1)	1.3501 (5)	0.8030 (2)	0.107 (2)
C11	0.600 (1)	1.3217 (5)	0.8317 (2)	0.109 (2)
C12	0.5537 (9)	1.2309 (5)	0.8563 (2)	0.081 (2)
C13	0.8621 (6)	0.6313 (4)	0.8876 (1)	0.047 (1)
C14	0.7585 (9)	0.5829 (4)	0.9174 (1)	0.074 (2)
C15	0.809 (1)	0.4718 (5)	0.9287 (2)	0.097 (2)
C16	0.9595 (9)	0.4136 (4)	0.9110 (2)	0.086 (2)
C17	1.0653 (9)	0.4646 (4)	0.8819 (2)	0.086 (2)
C18	1.0174 (8)	0.5754 (4)	0.8693 (1)	0.063 (1)
C19	0.8172 (7)	0.9431 (3)	0.8104 (1)	0.049 (1)
C20	0.7465 (8)	1.0247 (4)	0.7853 (1)	0.057 (1)
C21	0.8682 (8)	1.0593 (4)	0.7549 (1)	0.066 (1)
C22	1.0565 (8)	1.0119 (4)	0.7499 (1)	0.073 (2)
C23	1.1255 (8)	0.9302 (5)	0.7755 (1)	0.075 (2)
C24	1.0067 (8)	0.8962 (4)	0.8063 (1)	0.060 (1)

Table 4. *Geometric parameters* (Å, °) *for* (II)

S1—P1	1.900 (2)	P1—O1	1.600 (3)
S2—S3	2.070 (1)	P1—O2	1.567 (3)
S2—P1	2.075 (2)	P2—O3	1.588 (3)
S3—P2	2.076 (2)	P2—O4	1.582 (3)
S4—P2	1.898 (2)		
S3—S2—P1	101.38 (6)	O1—P1—O2	96.5 (2)
S2—S3—P2	104.26 (6)	S3—P2—S4	107.92 (8)
S1—P1—S2	108.39 (7)	S3—P2—O3	107.6 (1)
S1—P1—O1	118.1 (1)	S3—P2—O4	103.6 (1)
S1—P1—O2	120.8 (1)	S4—P2—O3	117.2 (1)
S2—P1—O1	106.3 (1)	S4—P2—O4	119.4 (1)
S2—P1—O2	105.2 (1)	O3—P2—O4	99.9 (2)
P1—S2—S3—P2	112.03 (7)	S2—S3—P2—S4	−175.98 (7)
S3—S2—P1—S1	−179.27 (7)		

The title compounds were obtained from iodine oxidation of the corresponding anions, $Ph_2PS_2^-$, $(PhO)_2PS_2^-$ (Maier, 1965), and crystallized from ethanol. Backgrounds were obtained from analysis of the scan profile (Blessing, Coppens & Becker, 1974). Data collection: CAD-4 (Enraf-Nonius, 1977). Cell refinement: CAD-4. Data reduction: *MolEN PROCESS* (Fair, 1990). Program(s) used to solve structure: direct methods *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Program(s) used to refine structure: *MolEN LSFM*. Molecular graphics: *ORTEP* (Johnson, 1976). Software used to prepare material for publication: *MolEN*.

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71242 (44 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR1049]

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18-Crown-6–Bis(methyl 10-camphorsulfonate) (1/2)

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Abstract

The 18-crown-6 (1,4,7,10,13,16-hexaoxacyclooctadecane) molecule has a regular conformation with approximate D_{3d} symmetry. The guest molecules (methyl 7,7-dimethyl-2-oxobicyclo[2.2.1]heptane-1-methanesulfonate) are coordinated above and below

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the crown in such a way that the dipoles are compensated. There are close contacts between the O—methyl C atom of each sulfonate and the O atoms of the crown ring [C···O distances from 3.264 (7) to 3.801 (7) Å].

Comment

The complex was prepared in 86% yield by mixing methyl 10-camphorsulfonate and 18-crown-6 in tetrahydrofuran followed by the addition of diethyl ether. The solution was cooled and the complex recrystallized from diethyl ether (melting point 326–327 K).

In this 1:2 complex, the two sulfonate molecules are coordinated above and below the crown and the methyl group of each sulfonate molecule yields C—H···O contacts to the O atoms of the crown. The 18-crown-6 molecule has approximate D_{3d} symmetry with mean O—C—C—O angles of 69.4 (4)° and C—C—O—C angles of 176.3 (6)°.

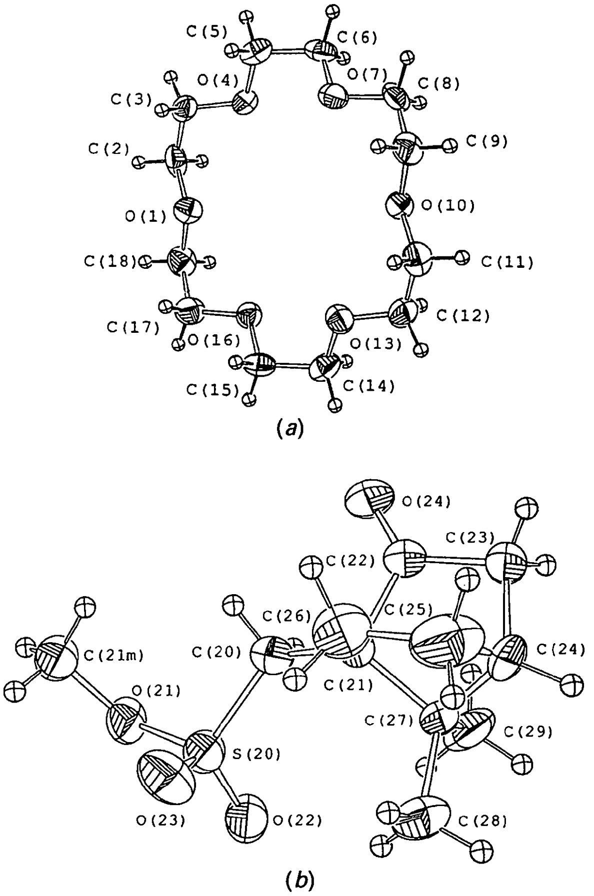

Fig. 1. *ORTEP* (Johnson, 1965) views of (*a*) 18-crown-6 and (*b*) $C_{11}H_{18}SO_4$ with the numbering schemes adopted. Ellipsoids are drawn at the 50% probability level and H atoms are represented by arbitrary spheres.

Small neutral molecules can form adducts with crown ethers if they present one or both of the following structural features: a significant dipole moment; *X*—H bonds (*X* = O, N, C) capable of forming hydrogen bonds with the O atoms of the crown in a complementary spatial arrangement (Grootenhuis & Kollman, 1989; Uiterwijk, Harkema & Feil, 1987). Complexes between crown ethers and neutral organic guests exhibit some of the properties characteristic of biological regulatory processes and enzyme catalysis (Vögtle, Müller & Watson, 1985).

The study of crown complexes involving C—H acidic guest molecules may help to promote understanding of the significance of C—H···O hydrogen bonds in organic crystals (Desiraju, 1991).

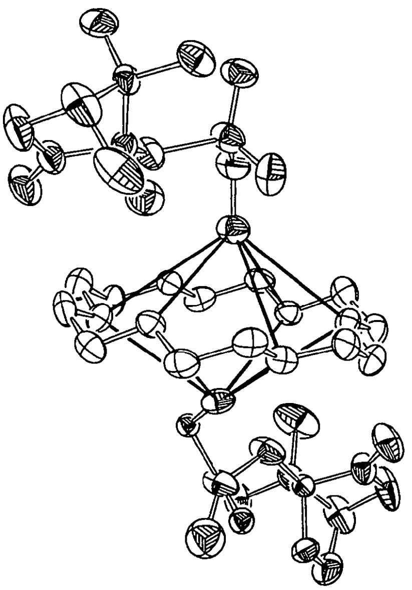

Fig. 2. *ORTEP* view of the association of the 2:1 complex. H atoms have been omitted and only the *X* set of isotropically refined O atoms is represented for clarity. Short contacts are represented by thin bonds.

Experimental

Crystal data

$C_{12}H_{24}O_6.2C_{11}H_{18}SO_4$
M_r = 756.96
Monoclinic
$P2_1$
a = 7.764 (3) Å
b = 18.692 (8) Å
c = 13.775 (6) Å
β = 100.05 (3)°
V = 1968.4 (14) Å^3
Z = 2
D_x = 1.277 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.70930 Å
Cell parameters from 25 reflections
θ = 9–10.25°
μ = 0.17 mm^{-1}
T = 243 K
Parallelepiped
0.49 × 0.42 × 0.25 mm
White

Data collection

Enraf-Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3854 measured reflections
3575 independent reflections
2661 observed reflections [$I > 3\sigma(I)$]
$R_{\rm int} = 0.017$
$\theta_{\rm max} = 25°$
$h = -9 \rightarrow 9$
$k = 0 \rightarrow 22$
$l = 0 \rightarrow 16$
5 standard reflections
frequency: 60 min
intensity variation: 2%

Refinement

Refinement on F
Final $R = 0.054$
$wR = 0.059$
$S = 2.31$
2661 reflections
461 parameters
$w = 1/[\sigma^2(F_o)+0.0002(F_o)^2]$
$(\Delta/\sigma)_{\rm max} < 0.10$
$\Delta\rho_{\rm max} = 0.53$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.38$ e Å^{-3}
Secondary-extinction coefficient: 0.29 (5)
Atomic scattering factors from Cromer & Mann (1968) for non-H atoms; Stewart, Davidson & Simpson (1965) for H atoms.

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

$B_{\rm eq}$ is the mean of the principal axes of the thermal ellipsoid.

	x	y	z	$B_{\rm eq}$ or $B_{\rm iso}$*
O(1)	0.2875 (5)	0.2815 (2)	0.3749 (3)	3.2 (2)
C(2)	0.4117 (9)	0.2888 (3)	0.4646 (4)	3.8 (3)
C(3)	0.5760 (8)	0.3168 (3)	0.4420 (4)	3.1 (2)
O(4)	0.5522 (5)	0.3886 (2)	0.4090 (3)	3.1 (2)
C(5)	0.7031 (8)	0.4165 (4)	0.3796 (4)	3.6 (3)
C(6)	0.6637 (7)	0.4882 (3)	0.3323 (5)	3.4 (3)
O(7)	0.5578 (5)	0.4781 (2)	0.2368 (3)	3.2 (2)
C(8)	0.5209 (8)	0.5436 (3)	0.1852 (5)	3.6 (3)
C(9)	0.4062 (8)	0.5300 (4)	0.0901 (5)	3.7 (3)
O(10)	0.2407 (5)	0.5079 (2)	0.1066 (3)	2.9 (2)
C(11)	0.1176 (9)	0.5014 (4)	0.0192 (4)	3.8 (3)
C(12)	−0.0496 (8)	0.4738 (4)	0.0434 (4)	3.9 (3)
O(13)	−0.0271 (5)	0.4021 (2)	0.0765 (3)	3.2 (2)
C(14)	−0.1812 (8)	0.3723 (4)	0.1018 (4)	3.6 (3)
C(15)	−0.1395 (7)	0.3005 (4)	0.1497 (4)	3.4 (3)
O(16)	−0.0361 (5)	0.3099 (2)	0.2440 (3)	2.9 (2)
C(17)	0.0098 (8)	0.2436 (4)	0.2905 (4)	3.7 (3)
C(18)	0.1212 (9)	0.2553 (4)	0.3886 (4)	4.0 (3)
S(20)	0.2184 (2)	0.5572 (1)	0.4854 (1)	3.9 (1)
O(21)	0.0985 (5)	0.5151 (3)	0.4038 (3)	4.1 (2)
O(22)	0.1113 (6)	0.6033 (3)	0.5288 (3)	4.8 (2)
O(23)	0.3557 (6)	0.5915 (3)	0.4401 (3)	4.9 (2)
O(24)	0.4523 (7)	0.3803 (2)	0.6851 (3)	4.9 (2)
C(21*m*)	0.1665 (8)	0.4692 (4)	0.3328 (4)	3.7 (3)
C(20)	0.3111 (8)	0.4907 (3)	0.5665 (5)	3.7 (3)
C(21)	0.4648 (7)	0.5077 (3)	0.6483 (4)	2.7 (3)
C(22)	0.4997 (9)	0.4401 (3)	0.7110 (4)	3.7 (3)
C(23)	0.6054 (12)	0.4621 (4)	0.8084 (5)	6.0 (4)
C(24)	0.6350 (10)	0.5414 (4)	0.7894 (5)	5.2 (3)
C(25)	0.7586 (9)	0.5446 (4)	0.7129 (7)	6.1 (4)
C(26)	0.6469 (9)	0.5198 (4)	0.6198 (5)	4.6 (4)
C(27)	0.4616 (8)	0.5651 (3)	0.7292 (4)	2.9 (3)
C(28)	0.4665 (9)	0.6426 (3)	0.6943 (5)	4.3 (3)
C(29)	0.3037 (10)	0.5570 (4)	0.7803 (5)	4.9 (4)
S(30)	0.3261 (2)	0.2395 (1)	0.0065 (1)	4.9 (1)
O(31*X*)	0.2808 (11)	0.2565 (5)	0.1164 (6)	2.3 (2)*
O(32*X*)	0.4762 (12)	0.2599 (5)	−0.0129 (7)	2.8 (2)*
O(33*X*)	0.2753 (13)	0.1581 (6)	0.0033 (7)	3.7 (2)*
O(31*Y*)	0.3363 (13)	0.2605 (6)	0.1129 (7)	3.6 (2)*
O(32*Y*)	0.4434 (18)	0.1957 (8)	−0.0205 (10)	7.0 (3)*
O(33*Y*)	0.4599 (15)	0.3024 (6)	−0.0291 (8)	5.1 (3)*
O(31*Z*)	0.4361 (32)	0.2856 (14)	0.0924 (17)	5.6 (6)*
O(32*Z*)	0.1985 (37)	0.1978 (16)	0.0619 (21)	7.0 (7)*
O(33*Z*)	0.3641 (19)	0.1684 (8)	−0.0007 (10)	0.9 (3)*
O(34)	0.0970 (7)	0.3991 (2)	−0.1931 (4)	5.6 (3)
C(31*m*)	0.3542 (8)	0.3294 (4)	0.1545 (4)	3.6 (3)
C(30)	0.1441 (10)	0.2765 (4)	−0.0628 (5)	5.3 (4)
C(31)	0.1125 (7)	0.2697 (3)	−0.1742 (4)	2.5 (2)
C(32)	0.0675 (9)	0.3402 (3)	−0.2268 (4)	3.8 (3)
C(33)	−0.0249 (13)	0.3226 (4)	−0.3292 (5)	6.4 (4)
C(34)	−0.0312 (10)	0.2420 (4)	−0.3275 (5)	5.1 (3)
C(35)	0.1555 (11)	0.2148 (4)	−0.3285 (5)	5.2 (4)
C(36)	0.2550 (8)	0.2369 (4)	−0.2265 (4)	3.5 (3)
C(37)	−0.0520 (8)	0.2259 (3)	−0.2185 (5)	3.8 (3)
C(38)	−0.0395 (10)	0.1455 (4)	−0.1949 (6)	5.3 (4)
C(39)	−0.2200 (10)	0.2540 (5)	−0.1926 (8)	7.6 (5)

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

O(1)—C(2)	1.435 (7)	C(24)—C(25)	1.545 (12)
O(1)—C(18)	1.423 (8)	C(24)—C(27)	1.518 (9)
C(2)—C(3)	1.462 (10)	C(25)—C(26)	1.491 (10)
C(3)—O(4)	1.420 (7)	C(27)—C(28)	1.530 (9)
O(4)—C(5)	1.405 (7)	C(27)—C(29)	1.524 (9)
C(5)—C(6)	1.498 (9)	S(30)—O(31*X*)	1.644 (8)
C(6)—O(7)	1.438 (7)	S(30)—O(32*X*)	1.298 (10)
O(7)—C(8)	1.419 (7)	S(30)—O(33*X*)	1.569 (11)
C(8)—C(9)	1.471 (9)	S(30)—O(31*Y*)	1.506 (10)
C(9)—O(10)	1.406 (7)	S(30)—O(32*Y*)	1.325 (15)
O(10)—C(11)	1.405 (7)	S(30)—O(33*Y*)	1.698 (12)
C(11)—C(12)	1.488 (10)	S(30)—O(31*Z*)	1.588 (25)
C(12)—O(13)	1.417 (8)	S(30)—O(32*Z*)	1.56 (3)
O(13)—C(14)	1.418 (8)	S(30)—O(33*Z*)	1.367 (15)
C(14)—C(15)	1.505 (9)	S(30)—C(30)	1.707 (7)
C(15)—O(16)	1.413 (7)	O(31*X*)—C(31*m*)	1.534 (11)
O(16)—C(17)	1.411 (8)	O(31*Y*)—C(31*m*)	1.406 (12)
C(17)—C(18)	1.488 (8)	O(31*Z*)—C(31*m*)	1.41 (3)
S(20)—O(21)	1.544 (4)	O(34)—C(32)	1.201 (8)
S(20)—O(22)	1.401 (4)	C(30)—C(31)	1.517 (8)
S(20)—O(23)	1.473 (5)	C(31)—C(32)	1.515 (8)
S(20)—C(20)	1.741 (6)	C(31)—C(36)	1.548 (8)
O(21)—C(21*m*)	1.467 (8)	C(31)—C(37)	1.550 (8)
O(24)—C(22)	1.211 (8)	C(32)—C(33)	1.503 (9)
C(20)—C(21)	1.525 (8)	C(33)—C(34)	1.509 (10)
C(21)—C(22)	1.528 (8)	C(34)—C(35)	1.538 (11)
C(21)—C(26)	1.548 (9)	C(34)—C(37)	1.566 (10)
C(21)—C(27)	1.549 (8)	C(35)—C(36)	1.538 (9)
C(22)—C(23)	1.503 (9)	C(37)—C(38)	1.537 (9)
C(23)—C(24)	1.531 (10)	C(37)—C(39)	1.506 (10)
C(2)—O(1)—C(18)	114.0 (4)	C(24)—C(27)—C(29)	115.3 (5)
O(1)—C(2)—C(3)	109.3 (5)	C(28)—C(27)—C(29)	107.7 (5)
C(2)—C(3)—O(4)	109.7 (5)	O(31*X*)—S(30)—O(32*X*)	118.5 (5)
C(3)—O(4)—C(5)	112.0 (4)	O(31*X*)—S(30)—O(33*X*)	96.9 (5)
O(4)—C(5)—C(6)	109.8 (5)	O(31*X*)—S(30)—C(30)	98.4 (4)
C(5)—C(6)—O(7)	108.8 (5)	O(32*X*)—S(30)—O(33*X*)	120.7 (6)
C(6)—O(7)—C(8)	112.3 (4)	O(32*X*)—S(30)—C(30)	116.8 (5)
O(7)—C(8)—C(9)	109.6 (5)	O(33*X*)—S(30)—C(30)	101.4 (5)
C(8)—C(9)—O(10)	109.6 (5)	O(31*Y*)—S(30)—O(32*Y*)	120.9 (7)
C(9)—O(10)—C(11)	113.1 (4)	O(31*Y*)—S(30)—O(33*Y*)	99.7 (6)
O(10)—C(11)—C(12)	109.1 (5)	O(31*Y*)—S(30)—C(30)	109.6 (5)
C(11)—C(12)—O(13)	109.7 (5)	O(32*Y*)—S(30)—O(33*Y*)	82.1 (8)
C(12)—O(13)—C(14)	112.9 (5)	O(32*Y*)—S(30)—C(30)	129.5 (6)
O(13)—C(14)—C(15)	109.4 (5)	O(33*Y*)—S(30)—C(30)	92.6 (5)
C(14)—C(15)—O(16)	109.7 (5)	O(31*Z*)—S(30)—O(32*Z*)	102.2 (14)
C(15)—O(16)—C(17)	111.5 (5)	O(31*Z*)—S(30)—O(33*Z*)	119.1 (11)
O(16)—C(17)—C(18)	110.1 (5)	O(31*Z*)—S(30)—C(30)	118.8 (10)
O(1)—C(18)—C(17)	109.1 (5)	O(32*Z*)—S(30)—O(33*Z*)	73.4 (13)
O(21)—S(20)—O(22)	107.3 (3)	O(32*Z*)—S(30)—C(30)	86.6 (11)
O(21)—S(20)—O(23)	107.6 (3)	O(33*Z*)—S(30)—C(30)	121.4 (6)
O(21)—S(20)—C(20)	103.4 (3)	S(30)—O(31*X*)—C(31*m*)	111.1 (6)
O(22)—S(20)—O(23)	116.2 (3)	S(30)—O(31*Y*)—C(31*m*)	128.4 (8)
O(22)—S(20)—C(20)	112.0 (3)	S(30)—O(31*Z*)—C(31*m*)	121.7 (17)
O(23)—S(20)—C(20)	109.5 (3)	S(30)—C(30)—C(31)	120.2 (5)
S(20)—O(21)—C(21*m*)	122.8 (4)	C(30)—C(31)—C(32)	113.2 (5)
S(20)—C(20)—C(21)	120.5 (4)	C(30)—C(31)—C(36)	120.1 (5)
C(20)—C(21)—C(22)	106.5 (5)	C(30)—C(31)—C(37)	114.8 (5)
C(20)—C(21)—C(26)	118.3 (5)	C(32)—C(31)—C(36)	104.3 (5)

C(20)—C(21)—C(27)	124.5 (5)	C(32)—C(31)—C(37)	99.5 (4)
C(22)—C(21)—C(26)	100.6 (5)	C(36)—C(31)—C(37)	102.4 (4)
C(22)—C(21)—C(27)	100.9 (4)	O(34)—C(32)—C(31)	126.8 (5)
C(26)—C(21)—C(27)	102.2 (5)	O(34)—C(32)—C(33)	126.2 (6)
O(24)—C(22)—C(21)	125.6 (5)	C(31)—C(32)—C(33)	106.9 (5)
O(24)—C(22)—C(23)	127.3 (6)	C(32)—C(33)—C(34)	102.4 (5)
C(21)—C(22)—C(23)	107.1 (5)	C(33)—C(34)—C(35)	107.3 (7)
C(22)—C(23)—C(24)	100.7 (5)	C(33)—C(34)—C(37)	102.5 (5)
C(23)—C(24)—C(25)	106.5 (6)	C(35)—C(34)—C(37)	101.6 (5)
C(23)—C(24)—C(27)	103.3 (6)	C(34)—C(35)—C(36)	103.1 (5)
C(25)—C(24)—C(27)	102.3 (5)	C(31)—C(36)—C(35)	104.2 (5)
C(24)—C(25)—C(26)	103.7 (6)	C(31)—C(37)—C(34)	93.5 (5)
C(21)—C(26)—C(25)	104.3 (6)	C(31)—C(37)—C(38)	114.6 (5)
C(21)—C(27)—C(24)	93.9 (4)	C(31)—C(37)—C(39)	114.3 (6)
C(21)—C(27)—C(28)	115.2 (5)	C(34)—C(37)—C(38)	112.1 (5)
C(21)—C(27)—C(29)	112.3 (5)	C(34)—C(37)—C(39)	113.5 (6)
C(24)—C(27)—C(28)	112.2 (5)	C(38)—C(37)—C(39)	108.4 (6)

The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1986) and difference Fourier synthesis using *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989) and refined by block-diagonal least squares on *F*. Peaks around the S(30) atom, for one of the methyl 10-camphorsulfonate moieties, were grouped into three sets of O atoms defining reasonable geometry. The population of each group gave refined occupancy factors (fixed in the final least-squares cycle) of 0.40, 0.40 and 0.20 for the *X*, *Y* and *Z* sets, respectively. These O atoms were refined isotropically with all other non-H atoms being refined anisotropically. In the final cycles, H-atom coordinates were fixed (C—H = 0.95 Å) and their isotropic temperature factors were set to the $U_{\rm iso}$ value of the C atom to which they were attached. Note that no H atoms were found for C(31*m*). Real and imaginary parts of the anomalous-dispersion correction were taken from Cromer & Liberman (1970).

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55914 (29 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CD1024]

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Structure of an α-(Methyldiphenylsilyl)-γ-lactone

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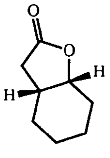
Abstract

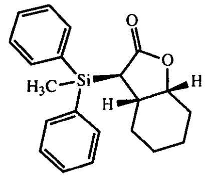
In the title compound, 3-methyl-3-(methyldiphenylsilyl)-3a,4,5,6,7,7a-hexahydro-1-benzofuran-2(3*H*)-one, the diphenylmethylsilyl group is *cis* to the cyclohexane ring, which is *cis*-fused to the lactone ring. The Si—C(lactone) bond length is 1.922 (2) Å, the Si—C(methyl) bond length is 1.866 (3) Å and the C(methyl)—Si—C—C(methyl) torsion angle is 152.8 (2)°.

Comment

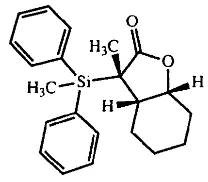
In the course of a study of the utility of α-(methyldiphenylsilyl)-γ-lactones in synthesis, we prepared the silylated lactone (2) (Betancourt de Perez, Fuentes, Larson, Barnes & Heeg, 1986). This material was formed by exclusive attack of methyldiphenylchlorosilane on the β-face of the lithium enolate of the lactone (1) (Grieco & Miyashita, 1974). We were interested in determining if this phenomenon of β-face attack would permit the placement of the bulky methyldiphenylsilyl group on the α-face of the molecule. Treatment of silylated lactone (2) with lithium diisopropylamide followed by methyl iodide yields (3). The crystal structure of title product (3) demonstrates that, not only can the bulky group occupy the α-position in (3), but also that the approach of an electrophile to the lithium enolate of lactone (1) is from the β-face, even under conditions which would seem to favor attack from the α-face.



(1)



(2)



(3)

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