(1'α,5'α,6'β,7'α,8'β)-Tetrahydrodispiro[furan-2(3*H*),6'-[2,4,10]trioxaadamantane-8',2''(3''*H*)-furan]-9'-one (6). To a cooled (-78 °C) CH_2Cl_2 solution (50 mL) of oxalyl chloride (0.86 mL, 9.86 mmol) were added DMSO (1.11 mL, 15.6 mmol) in CH_2Cl_2 (5 mL) and the alcohol (1.01 g, 3.74 mmol) dissolved in CH_2Cl_2 (15 mL) within 5 min. The mixture was stirred for 2 h at -78 °C, treated with triethylamine (2.72 mL, 19.5 mmol), and warmed to rt for 1 h before being washed with water and extracted with CH_2Cl_2 (2 x 50 mL). The combined organic phases were dried and concentrated in vacuo to leave a residue that was purified by chromatography on silica gel (elution with 50% ethyl acetate in hexanes containing 5% MeOH). There was isolated 974 mg (97%) of **6** as a white solid, mp 244-246 °C (from 5% CH_2Cl_2 in hexanes); IR (film, cm^{-1}) 1767, 1462, 1378, 1267; ^1H NMR (300 MHz, $CDCl_3$) δ 5.61 (s, 1 H), 4.05-3.90 (m, 6 H), 3.70 (m, 1 H), 2.55-2.40 (m, 2 H), 2.30-2.15 (m, 2 H), 2.05-1.85 (m, 4 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 200.2, 102.5, 82.9, 80.1, 78.4, 70.0, 34.9, 24.3; HRMS *m/z* (M^+) calcd 268.0947, obsd 268.0924.

(1α,5α,6α,7α,8β,9β)-Hexahydrotrispiro[2,4,10-trioxaadamantane-6,2'(3'*H*);8,2''(3''*H*);9,2'''(3'''*H*)-trisfuran] (7). To a cooled (-78 °C) solution of **6** (112 mg, 0.418 mmol) in THF (10 mL) was added 0.26 N Normant reagent (3.2 mL, 0.83 mmol). The reaction mixture was warmed to rt for 2 h, quenched with saturated NH_4Cl solution, and filtered. The filtrate was concentrated and the residue was purified by chromatography on silica gel (elution with 25% ethyl acetate in hexanes) to afford the diol (~10 :1) as a colorless liquid.

This diol in CH_2Cl_2 (10 mL) was treated with *p*-toluenesulfonyl chloride (159 mg, 0.834 mmol), triethylamine (0.23 mL, 1.6 mmol), and DMAP (10 mg) at rt for 15 h and refluxed for 1 d. The reaction mixture was concentrated in vacuo and the residue was purified by chromatography on silica gel (elution with 50% ethyl acetate in hexanes) to afford 58 mg (45%) of **7** as a colorless oil; IR (film, cm^{-1}) 1434, 1372, 1280, 1157; ^1H NMR (300 MHz, $CDCl_3$) δ 5.36 (s, 1 H), 4.05-3.85 (m, 4 H), 3.75-3.60 (m, 4 H), 3.55 (m, 1 H), 2.45-2.25 (m, 2 H), 2.25-2.15 (m, 2 H), 2.10-2.00 (m, 2 H), 2.00-1.85 (m, 4 H), 1.80-1.70 (m, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ

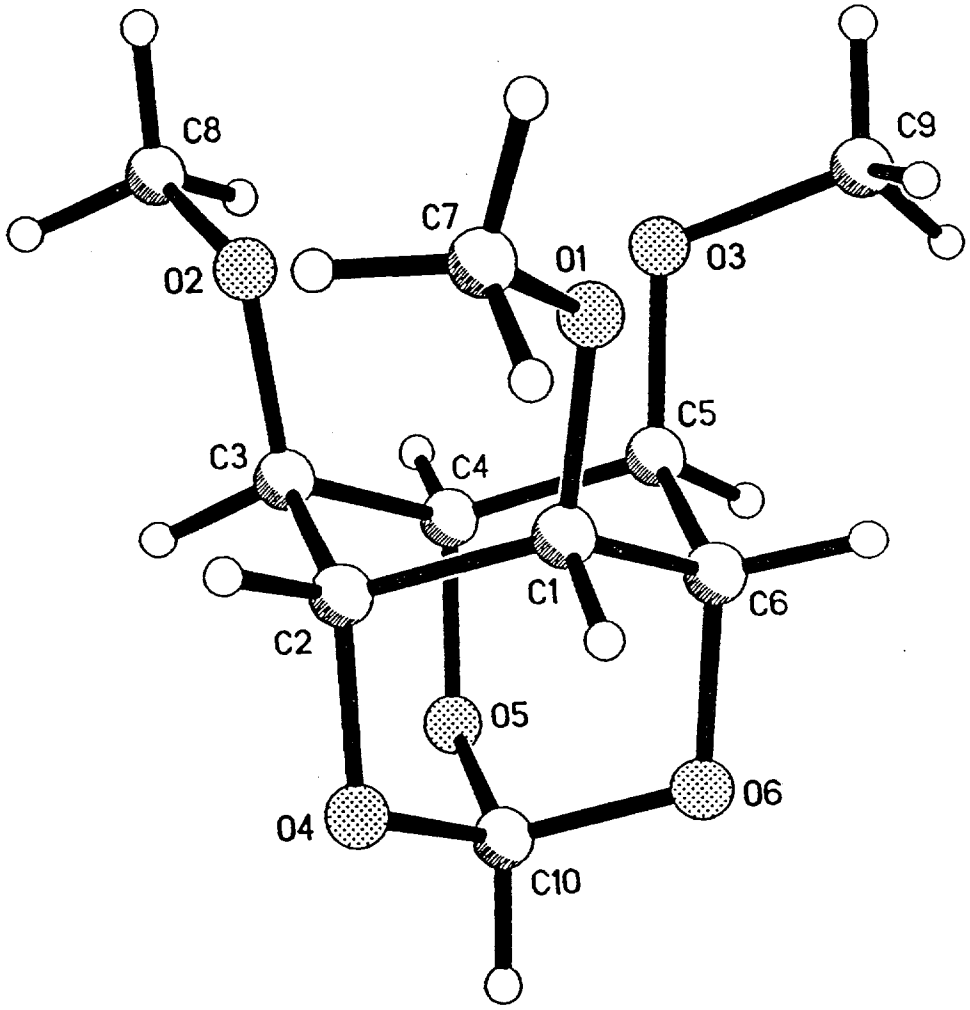
102.1, 80.8, 76.6, 74.4, 71.0, 69.2, 63.2, 35.9, 33.3, 25.8, 23.9; HRMS *m/z* (M^+) calcd 310.1416, obsd 310.1430.

(1α,5α,6β,7α,8β,9β)-Hexahydrotrispiro[2,4,10-trioxaadamantane-6,2'(3'*H*);8,2''(3''*H*);9,2'''(3'''*H*)-trisfuran] (2). To a cooled (-78 °C) solution of **6** (170 mg, 0.634 mmol), which was pre-complexed with $LiClO_4$ (337 mg, 3.17 mmol) in THF (10 mL) for 2 h at rt, was added 0.26 N Normant reagent (7.3 mL, 1.90 mmol). The reaction mixture was warmed to rt for 15 h, quenched with saturated NH_4Cl solution, and filtered. The filtrate was concentrated and the residue was purified by chromatography on silica gel (elution with 50% ethyl acetate in hexanes containing 5% MeOH) to afford the diol.

This diol in CH_2Cl_2 (10 mL) was treated with *p*-toluenesulfonyl chloride (255 mg, 1.34 mmol), triethylamine (0.37 mL, 2.7 mmol), and DMAP (10 mg) at rt for 15 h. The reaction mixture was concentrated in vacuo and the residue was purified by chromatography on silica gel (elution with 20-50% ethyl acetate in hexanes) to afford 262 mg of the monotosylate as a white solid.

To a benzene (10 mL) solution of the monotosylate was added 0.5 N potassium hexamethyldisilazide (2.2 mL, 1.1 mmol) at 0 °C. After 30 min, the reaction mixture was warmed to rt, stirred for 15 h, quenched with deionized H_2O (10 mL), and extracted with CH_2Cl_2 (3 x 20 mL). The combined organic phases were washed with deionized H_2O (10 mL) and concentrated in vacuo without drying to give 144 mg (73% for 3 steps) of **2** as a white solid, mp >280 °C (from 10% CH_2Cl_2 in hexanes); IR (film, cm^{-1}) 1440, 1350, 1250, 1125; ^1H NMR (300 MHz, $CDCl_3$) δ 5.37 (s, 1 H), 3.95 (t, J = 6.8 Hz, 6 H), 3.51 (s, 3 H), 2.26 (t, J = 7.3 Hz, 6 H), 1.91-1.80 (m, 6 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 102.4, 80.0, 75.9, 69.4, 36.3, 23.9; HRMS *m/z* (M^+) calcd 310.1416, obsd 310.1427.

Anal. Calcd for $C_{12}H_{22}O_6$: C, 61.92; H, 7.14. Found: C, 61.68; H, 7.06.



Perspective plot of **9** in the solid state

Table 1. Crystal Data and Structure Refinement for **9**.

Color / Shape	colorless / fragment
Empirical formula	$C_{10}H_{16}O_6$
Formula weight	232.23
Temperature	173(2) K
Crystal system	Monoclinic
Space group	C2/c
Unit cell dimensions	a = 10.6460(2) Å $\alpha = 90^{\circ}$
(4966 reflections	b = 8.250 Å $\beta = 90.264(1)^{\circ}$
in full θ range)	c = 24.1836(1) Å $\gamma = 90^{\circ}$
Volume	2123.96(4) $Å^3$
Z	8
Density (calculated)	1.452 Mg/m^3
Absorption coefficient	0.121 mm^{-1}
Diffractometer / scan	Siemens SMART / CCD area detector
Radiation / wavelength	MoKα (graphite monochrom.) / 0.71073 Å
F(000)	992
Crystal size	0.30 x 0.25 x 0.15 mm
θ range for data collection	1.68 to 27.91°
Index ranges	$-13 \le h \le 7$, $-10 \le k \le 10$, $-31 \le \ell \le 31$
Reflections collected	6536
Independent / observed refls.	2460(R_{int} = 0.0182) / 2297([I>2σ(I)])
Absorption correction	SADABS[1]
Refinement method	Full-matrix least-squares on F^2
Computing	SHELXTL, Ver. 5[2]
Data / restraints / parameters	2452 / 0 / 149
Goodness-of-fit on F^2	0.922
SHELX-93 weight parameters	0.0859, 24.7281
Final R indices [I>2σ(I)]	R1 = 0.0855, wR2 = 0.2253
R indices (all data)	R1 = 0.0919, wR2 = 0.2530
Extinction coefficient	0.015(2)
Largest diff. peak and hole	0.418 and -0.385 $eÅ^{-3}$

Table 2. Atomic Coordinates [x 10^4] and Equivalent Isotropic Displacement Parameters [$Å^2$ x 10^3] for **9**.

Atom	x/a	y/b	z/c	U(eq)
O(1)	-2228(2)	1543(3)	6947(1)	28(1)
O(2)	-2871(2)	275(3)	5836(1)	27(1)
O(3)	-1274(2)	3081(3)	5951(1)	25(1)
O(4)	-331(2)	-1947(3)	6496(1)	28(1)
O(5)	515(2)	-603(3)	5744(1)	28(1)
O(6)	952(2)	285(3)	6632(1)	26(1)
C(1)	-1225(3)	453(4)	6877(1)	23(1)
C(2)	-1473(3)	-984(4)	6483(2)	25(1)
C(3)	-1699(3)	-512(4)	5877(1)	24(1)
C(4)	-560(3)	466(4)	5690(1)	24(1)
C(5)	-294(3)	1968(4)	6041(1)	22(1)
C(6)	-112(3)	1384(4)	6643(1)	22(1)
C(7)	-3209(4)	886(6)	7279(2)	38(1)
C(8)	-3281(4)	437(6)	5279(2)	40(1)
C(9)	-1033(4)	4630(5)	6192(2)	32(1)
C(10)	699(3)	-1076(4)	6297(2)	27(1)

U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 3. Bond Lengths [Å] and Angles [°] for **9**.

O(1)-C(1)	1.407(4)	O(1)-C(7)	1.426(5)
O(2)-C(3)	1.410(4)	O(2)-C(8)	1.421(5)
O(3)-C(5)	1.405(4)	O(3)-C(9)	1.427(4)
O(4)-C(10)	1.399(5)	O(4)-C(2)	1.452(4)
O(5)-C(10)	1.407(4)	O(5)-C(4)	1.449(4)
O(6)-C(10)	1.409(4)	O(6)-C(6)	1.451(4)
C(1)-C(6)	1.523(5)	C(1)-C(2)	1.543(5)
C(2)-C(3)	1.534(5)	C(3)-C(4)	1.527(5)
C(4)-C(5)	1.528(5)	C(5)-C(6)	1.544(5)

C(1)-O(1)-C(7)	112.5(3)	C(3)-O(2)-C(8)	112.2(3)
C(5)-O(3)-C(9)	113.0(3)	C(10)-O(4)-C(2)	111.6(3)
C(10)-O(5)-C(4)	111.1(3)	C(10)-O(6)-C(6)	111.2(2)
O(1)-C(1)-C(6)	108.4(3)	O(1)-C(1)-C(2)	116.0(3)
C(6)-C(1)-C(2)	106.8(3)	O(4)-C(2)-C(3)	106.7(3)
O(4)-C(2)-C(1)	105.4(3)	C(3)-C(2)-C(1)	114.8(3)
O(2)-C(3)-C(4)	116.1(3)	O(2)-C(3)-C(2)	108.6(3)
C(4)-C(3)-C(2)	107.2(3)	O(5)-C(4)-C(5)	107.4(3)
O(5)-C(4)-C(3)	106.3(3)	C(5)-C(4)-C(3)	114.1(3)
O(3)-C(5)-C(4)	108.0(3)	O(3)-C(5)-C(6)	116.1(3)
C(4)-C(5)-C(6)	107.0(3)	O(6)-C(6)-C(1)	107.5(3)
O(6)-C(6)-C(5)	105.8(3)	C(1)-C(6)-C(5)	114.4(3)
O(4)-C(10)-O(5)	111.3(3)	O(4)-C(10)-O(6)	111.1(3)
O(5)-C(10)-O(6)	110.5(3)		

Symmetry transformations used to generate equivalent atoms:

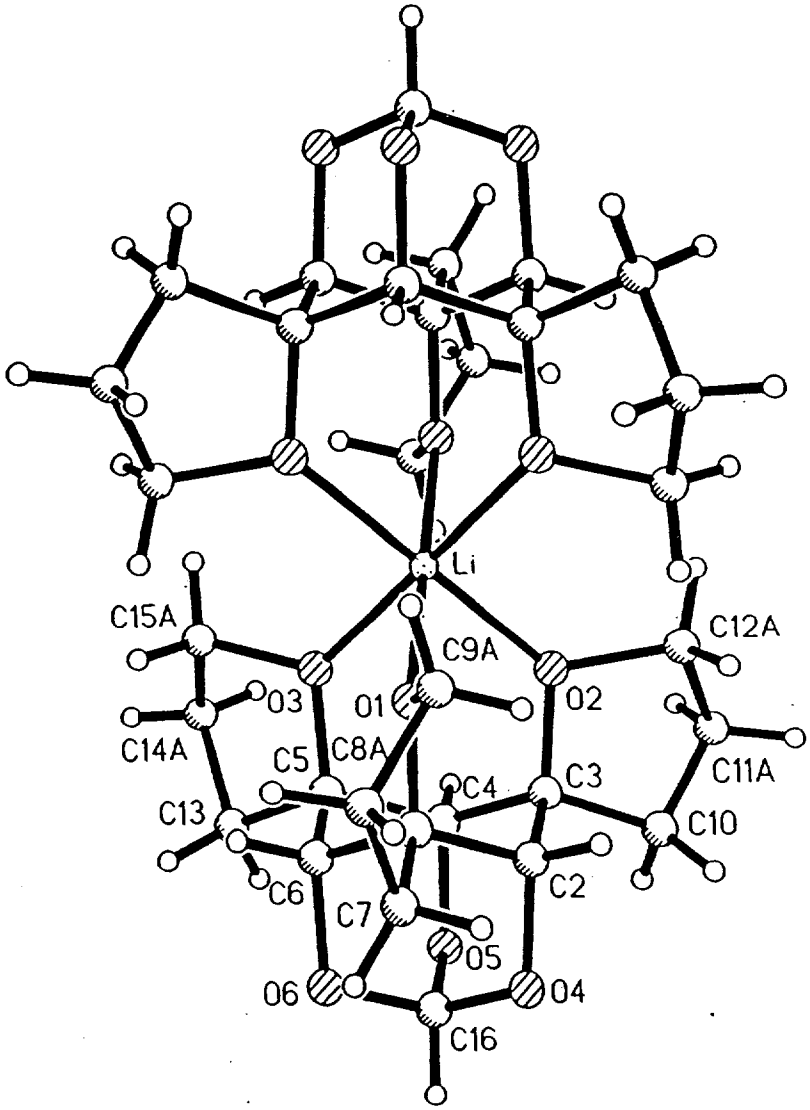
Table 4. Anisotropic Displacement Parameters [$Å^2 \times 10^3$] for **9**.

Atom	U11	U22	U33	U23	U13	U12
O(1)	29(1)	32(1)	24(1)	3(1)	6(1)	6(1)
O(2)	23(1)	33(1)	26(1)	-1(1)	-7(1)	1(1)
O(3)	27(1)	20(1)	29(1)	2(1)	-1(1)	4(1)
O(4)	30(1)	20(1)	32(1)	4(1)	-3(1)	4(1)
O(5)	30(1)	32(1)	22(1)	-2(1)	3(1)	12(1)
O(6)	21(1)	31(1)	26(1)	-2(1)	-6(1)	2(1)
C(1)	25(2)	27(2)	17(1)	2(1)	-1(1)	2(1)
C(2)	24(2)	23(2)	27(2)	4(1)	-2(1)	1(1)
C(3)	26(2)	21(2)	24(2)	-3(1)	-6(1)	1(1)
C(4)	28(2)	25(2)	18(1)	0(1)	0(1)	7(1)
C(5)	21(2)	24(2)	22(2)	3(1)	1(1)	1(1)
C(6)	21(2)	26(2)	20(1)	-3(1)	-3(1)	-1(1)
C(7)	28(2)	54(3)	32(2)	5(2)	7(2)	2(2)
C(8)	37(2)	49(3)	33(2)	8(2)	-12(2)	2(2)
C(9)	36(2)	24(2)	36(2)	-4(2)	5(2)	3(2)
C(10)	28(2)	25(2)	28(2)	-1(1)	-2(1)	7(1)

The anisotropic displacement factor exponent takes the form:
$-2\pi^2 [(ha^*)^2 U_{11} + ... + 2hka^*b^* U_{12}]$.

Table 5. Hydrogen Coordinates [x 10^4] and Isotropic Displacement Parameters [$Å^2$ x 10^3] for **9**.

Atom	x	y	z	U(eq)
H(1A)	-982(3)	13(4)	7248(1)	27
H(2A)	-2195(3)	-1642(4)	6621(2)	30
H(3A)	-1739(3)	-1528(4)	5652(1)	29
H(4A)	-669(3)	789(4)	5294(1)	28
H(5A)	509(3)	2468(4)	5913(1)	27
H(6A)	86(3)	2330(4)	6886(1)	27
H(7A)	-3817(17)	1740(11)	7365(12)	57
H(7B)	-2852(5)	456(38)	7623(7)	57
H(7C)	-3631(21)	12(29)	7076(6)	57
H(8A)	-4065(17)	1060(35)	5268(2)	60
H(8B)	-3425(30)	-640(6)	5120(5)	60
H(8C)	-2637(14)	1004(36)	5065(4)	60
H(9A)	-1621(19)	5427(9)	6039(9)	48
H(9B)	-170(10)	4962(17)	6109(11)	48
H(9C)	-1141(28)	4565(10)	6594(2)	48
H(10A)	1450(3)	-1803(4)	6312(2)	32



Perspective plot of **10** in the solid state

X-ray Data Collection, Structure Determination, and refinement for 10. A colorless single crystal of the title compound was mounted on a fiber and transferred to the goniometer. The space group was determined to be either the centric C2/c or the acentric Cc from the systematic absences. The structure was initially solved in C2/c where +/- gauche disorder in three ethylene groups and positional disorder of the anion were noted. An investigation of the acentric space group Cc produced a higher R value, correlations between refined parameters and the disorder was still present. The final refinements were carried out in the centric C2/c. A summary of data collection parameters is given in Table 6.

The perchlorate anion was positionally disordered about a two-fold axis. The Cl reside on the axis and the disorder was modeled with several partially occupied positions of the oxygen atoms. This included three oxygen atoms in general positions at 50% occupancy, one at 25% occupancy, one at 12.5% occupancy, and one oxygen atom on a special position at 25% occupancy (12.5% overall).

The ligand was also found to be disordered. The ethylene groups C8-C9, C11-C12,and C14-C15 were modeled with +/ gauche disorder. Two alternate positions were reined for each atom at 50% occupancy.

The hydrogen atoms were placed in calculated positions and allowed to ride on the bonded atom with B = 1.2*Ueqv (C). Refinement of nonhydrogen atoms was carried out with anisotropic temperature factors. The final values of the positional parameters are given in Table 7.

Table 6. Crystal Data and Structure Refinement for **10**.

Color / Shape	colorless / fragment
Empirical formula	$C_{32}H_{44}ClLiO_{16}$
Formula weight	727.06
Temperature	295(2) K
Crystal system	Monoclinic
Space group	C2/c
Unit cell dimensions	a = 15.1988(1) Å α = 90°
(4373 reflections	b = 13.9934(1) Å β = 99.951(2)°
in full θ range)	c = 15.7082(3) Å γ = 90°
Volume	3290.60(7) Å^3
Z	4
Density (calculated)	1.468 Mg/m^3
Absorption coefficient	0.194 mm^{-1}
Diffractometer / scan	Siemens SMART / CCD area detector
Radiation / wavelength	MoKα (graphite monochrom.) / 0.71073 Å
F(000)	1536
Crystal size	0.40 x 0.44 x 0.50 mm
θ range for data collection	1.99 to 25.00°
Index ranges	$-19 \le h \le 19$, $-17 \le k \le 13$, $-20 \le \ell \le 15$
Reflections collected	8365
Independent / observed refls.	2897(R_{int} = 0.0327) / 2272([I>2σ(I)])
Absorption correction	SADABS[1]
Refinement method	Full-matrix-block least-squares on F^2
Computing	SHELXTL, Ver. 5[2]
Data / restraints / parameters	2720 / 0 / 312
Goodness-of-fit on F^2	1.028
SHELX-93 weight parameters	0.0644, 2.9212
Final R indices [I>2σ(I)]	R1 = 0.0475, wR2 = 0.1223
R indices (all data)	R1 = 0.0635, wR2 = 0.1394
Extinction coefficient	0.0037(5)
Largest diff. peak and hole	0.230 and -0.202 eÅ^{-3}

Table 7. Atomic Coordinates [x 10^4] and Equivalent Isotropic Displacement Parameters [$Å^2$ x 10^3] for **10**.

Atom	x/a	y/b	z/c	U(eq)a
Cl	0	189(1)	2500	83(1)
Li	2500	2500	0	46(1)
O(1)	2867(1)	2556(1)	1314(1)	45(1)
O(2)	1199(1)	2449(1)	259(1)	51(1)
O(3)	2326(1)	3984(1)	101(1)	49(1)
O(4)	1080(1)	3399(1)	2422(1)	62(1)
O(5)	619(1)	4584(1)	1408(1)	64(1)
O(6)	2011(1)	4675(1)	2287(1)	58(1)
O(7A)b	16(4)	-494(4)	3103(4)	132(2)
O(8A)b	675(3)	774(4)	2846(4)	119(2)
O(7B)c	-398(15)	-154(12)	3335(10)	163(6)
O(8B)b	823(4)	689(4)	2494(6)	163(3)
O(7C)d	0	-736(9)	2500	130(6)
O(8C)e	394(18)	166(18)	3391(11)	109(8)
C(1)	2433(1)	3108(2)	1897(1)	42(1)
C(2)	1451(2)	2816(2)	1809(1)	48(1)
C(3)	882(1)	3005(2)	916(2)	48(1)
C(4)	972(2)	4072(2)	733(2)	49(1)
C(5)	1929(2)	4429(2)	769(1)	45(1)
C(6)	2449(1)	4168(2)	1668(1)	44(1)
C(7)	2966(2)	2903(2)	2812(1)	59(1)
C(8A)b	3765(3)	2379(4)	2661(3)	62(1)
C(9A)b	3407(4)	1824(4)	1826(3)	60(1)
C(8B)b	3618(3)	2118(3)	2650(3)	58(1)
C(9B)b	3772(3)	2297(4)	1752(3)	58(1)
C(10)	-102(2)	2704(2)	895(2)	75(1)
C(11A)b	-376(3)	2190(4)	125(4)	71(2)
C(12A)b	531(3)	1644(4)	106(3)	61(1)
C(11B)b	-156(4)	1801(5)	229(4)	89(2)
C(12B)b	537(4)	1942(5)	-299(4)	84(2)
C(13)	1965(2)	5518(2)	597(2)	72(1)

Table 7 (continued)

C(14A)b	2113(2)	5562(2)	-345(2)	75(3)
C(15A)b	2812(3)	4768(4)	-291(3)	59(1)
C(14B)b	2423(9)	5598(7)	-133(7)	104(3)
C(15B)b	2452(5)	4630(5)	-540(4)	87(2)
C(16)	1127(2)	4364(2)	2221(2)	63(1)

[a]U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor. [b]50% occupancy. [c]25% occupancy. [d]25% occupancy of a special position. E12.5% occupancy.

Table 8. Bond Lengths [Å] and Angles [°] for **10**.

Cl-O(7C)	1.294(12)	Cl-O(7A)	1.343(5)
Cl-O(8A)	1.352(4)	Cl-O(8C)	1.42(2)
Cl-O(8B)	1.436(5)	Cl-O(7B)	1.610(13)
Li-O(1)	2.0446(14)	Li-O(1)#2	2.0446(14)
Li-O(2)	2.088(2)	Li-O(2)#2	2.088(2)
Li-O(3)#2	2.103(2)	Li-O(3)	2.103(2)
O(1)-C(1)	1.441(2)	O(1)-C(9A)	1.463(5)
O(1)-C(9B)	1.473(5)	O(2)-C(12B)	1.407(5)
O(2)-C(3)	1.440(3)	O(2)-C(12A)	1.507(5)
O(3)-C(15B)	1.391(6)	O(3)-C(5)	1.439(3)
O(3)-C(15A)	1.511(5)	O(4)-C(16)	1.391(3)
O(4)-C(2)	1.449(3)	O(5)-C(16)	1.407(3)
O(5)-C(4)	1.457(3)	O(6)-C(16)	1.398(3)
O(6)-C(6)	1.455(3)	C(1)-C(6)	1.528(3)
C(1)-C(2)	1.530(3)	C(1)-C(7)	1.551(3)
C(2)-C(3)	1.539(3)	C(3)-C(4)	1.531(3)
C(3)-C(10)	1.549(3)	C(4)-C(5)	1.530(3)
C(5)-C(6)	1.538(3)	C(5)-C(13)	1.550(3)
C(7)-C(8A)	1.473(6)	C(7)-C(8B)	1.531(5)
C(8A)-C(9A)	1.539(7)	C(8B)-C(9B)	1.491(7)
C(10)-C(11A)	1.407(6)	C(10)-C(11B)	1.633(7)
C(11A)-C(12A)	1.581(7)	C(11B)-C(12B)	1.461(9)
C(13)-C(14B)	1.446(11)	C(13)-C(14A)	1.54
C(14A)-C(15A)	1.530(6)	C(14B)-C(15B)	1.501(12)

O(7A)-Cl-O(8A)	103.2(4)	O(8A)-Cl-O(8A)#1	105.3(5)
O(8C)-Cl-O(8B)#1	102.9(9)	O(1)-Li-O(1)#2	180.0
O(1)-Li-O(2)	84.65(6)	O(1)#2-Li-O(2)	95.35(6)
O(1)-Li-O(2)#2	95.35(6)	O(1)#2-Li-O(2)#2	84.65(6)
O(2)-Li-O(2)#2	180.0	O(1)-Li-O(3)#2	95.61(6)
O(1)#2-Li-O(3)#2	84.39(6)	O(2)-Li-O(3)#2	96.69(6)
O(2)#2-Li-O(3)#2	83.31(6)	O(1)-Li-O(3)	84.39(6)
O(1)#2-Li-O(3)	95.61(6)	O(2)-Li-O(3)	83.31(6)
O(2)#2-Li-O(3)	96.69(6)	O(3)#2-Li-O(3)	180.0
C(1)-O(1)-C(9A)	107.5(2)	C(1)-O(1)-C(9B)	109.2(2)
C(1)-O(1)-Li	125.89(12)	C(9A)-O(1)-Li	123.9(2)

Table 8 (continued)

C(9B)-O(1)-Li	122.1(2)	C(12B)-O(2)-C(3)	115.3(3)
C(3)-O(2)-C(12A)	102.7(2)	C(12B)-O(2)-Li	119.7(3)
C(3)-O(2)-Li	124.49(12)	C(12A)-O(2)-Li	128.9(2)
C(15B)-O(3)-C(5)	112.0(3)	C(5)-O(3)-C(15A)	106.2(2)
C(15B)-O(3)-Li	123.5(3)	C(5)-O(3)-Li	123.78(12)
C(15A)-O(3)-Li	127.5(2)	C(16)-O(4)-C(2)	110.8(2)
C(16)-O(5)-C(4)	109.9(2)	C(16)-O(6)-C(6)	110.1(2)
O(1)-C(1)-C(6)	110.1(2)	O(1)-C(1)-C(2)	110.2(2)
C(6)-C(1)-C(2)	107.1(2)	O(1)-C(1)-C(7)	105.5(2)
C(6)-C(1)-C(7)	111.8(2)	C(2)-C(1)-C(7)	112.2(2)
O(4)-C(2)-C(1)	106.0(2)	O(4)-C(2)-C(3)	106.7(2)
C(1)-C(2)-C(3)	114.9(2)	O(2)-C(3)-C(4)	109.9(2)
O(2)-C(3)-C(2)	110.8(2)	C(4)-C(3)-C(2)	106.6(2)
O(2)-C(3)-C(10)	106.2(2)	C(4)-C(3)-C(10)	112.2(2)
C(2)-C(3)-C(10)	111.2(2)	O(5)-C(4)-C(5)	106.3(2)
O(5)-C(4)-C(3)	106.6(2)	C(5)-C(4)-C(3)	115.2(2)
O(3)-C(5)-C(4)	110.3(2)	O(3)-C(5)-C(6)	110.6(2)
C(4)-C(5)-C(6)	106.9(2)	O(3)-C(5)-C(13)	105.5(2)
C(4)-C(5)-C(13)	112.1(2)	C(6)-C(5)-C(13)	111.4(2)
O(6)-C(6)-C(1)	106.7(2)	O(6)-C(6)-C(5)	106.0(2)
C(1)-C(6)-C(5)	114.8(2)	C(8A)-C(7)-C(1)	104.8(3)
C(8B)-C(7)-C(1)	103.2(2)	C(7)-C(8A)-C(9A)	102.1(4)
O(1)-C(9A)-C(8A)	101.4(4)	C(9B)-C(8B)-C(7)	104.4(3)
O(1)-C(9B)-C(8B)	101.4(3)	C(11A)-C(10)-C(3)	107.4(3)
C(3)-C(10)-C(11B)	99.5(3)	C(10)-C(11A)-C(12A)	97.7(4)
O(2)-C(12A)-C(11A)	101.6(4)	C(12B)-C(11B)-C(10)	107.3(5)
O(2)-C(12B)-C(11B)	102.9(5)	C(14B)-C(13)-C(5)	104.5(5)
C(14A)-C(13)-C(5)	102.84(14)	C(15A)-C(14A)-C(13)	97.7(2)
O(3)-C(15A)-C(14A)	99.5(3)	C(13)-C(14B)-C(15B)	108.8(6)
O(3)-C(15B)-C(14B)	105.1(5)	O(4)-C(16)-O(6)	111.8(2)
O(4)-C(16)-O(5)	112.1(2)	O(6)-C(16)-O(5)	111.7(2)

Symmetry transformations used to generate equivalent atoms:

#1 -x,y,-z+1/2 #2 -x+1/2,-y+1/2,-z

Table 9. Anisotropic Displacement Parameters [$Å^2 \times 10^3$] for **10**.

Atom	U11	U22	U33	U23	U13	U12
Cl	85(1)	45(1)	107(1)	0	-18(1)	0
O(1)	50(1)	52(1)	34(1)	-1(1)	6(1)	16(1)
O(2)	53(1)	54(1)	43(1)	-13(1)	4(1)	-1(1)
O(3)	64(1)	51(1)	36(1)	4(1)	22(1)	8(1)
O(4)	62(1)	86(1)	45(1)	-5(1)	29(1)	-2(1)
O(5)	60(1)	75(1)	62(1)	-12(1)	20(1)	23(1)
O(6)	68(1)	60(1)	49(1)	-20(1)	19(1)	1(1)
O(7A)	162(5)	108(4)	131(4)	66(3)	40(4)	3(4)
O(8A)	106(3)	96(3)	153(5)	-45(3)	17(3)	-32(3)
O(7B)	227(18)	177(14)	108(10)	2(9)	90(12)	-32(13)
O(8B)	99(4)	112(4)	261(9)	73(5)	-13(5)	-38(3)
O(7C)	155(14)	48(7)	199(18)	0	65(13)	0
O(8C)	135(18)	139(18)	45(9)	1(10)	-6(11)	65(16)
C(1)	49(1)	49(1)	29(1)	-2(1)	10(1)	3(1)
C(2)	53(1)	53(1)	39(1)	1(1)	19(1)	-3(1)
C(3)	41(1)	58(1)	47(1)	-7(1)	11(1)	1(1)
C(4)	48(1)	58(1)	43(1)	-4(1)	11(1)	17(1)
C(5)	58(1)	40(1)	41(1)	-1(1)	17(1)	8(1)
C(6)	50(1)	47(1)	38(1)	-8(1)	15(1)	-1(1)
C(7)	70(2)	71(2)	33(1)	2(1)	5(1)	3(1)
C(8A)	58(3)	72(3)	50(3)	12(3)	-8(2)	4(3)
C(9A)	71(3)	54(3)	52(3)	12(2)	3(2)	19(2)
C(8B)	71(3)	56(3)	42(2)	10(2)	-2(2)	8(2)
C(9B)	52(3)	72(3)	48(3)	7(2)	1(2)	18(2)
C(10)	44(1)	100(2)	81(2)	-5(2)	11(1)	-12(1)
C(11A)	43(2)	89(4)	78(4)	-20(3)	3(2)	-6(3)
C(12A)	74(3)	61(3)	50(3)	-19(2)	13(2)	-14(2)
C(11B)	76(4)	101(5)	85(4)	-15(4)	3(3)	-26(4)
C(12B)	59(3)	104(5)	83(4)	-20(4)	-5(3)	-19(3)
C(13)	103(2)	43(1)	71(2)	6(1)	21(2)	8(1)
C(14A)	125(8)	51(4)	55(4)	18(3)	33(4)	23(5)
C(15A)	63(3)	64(3)	55(3)	7(2)	28(2)	-4(2)
C(14B)	118(7)	82(6)	120(7)	23(5)	39(6)	-30(5)

Table 9 (continued)

C(15B)	118(5)	82(4)	64(4)	30(3)	30(4)	1(4)
C(16)	68(2)	76(2)	51(1)	-14(1)	26(1)	14(1)

The anisotropic displacement factor exponent takes the form:
$-2\pi^2 [(ha^*)^2 U_{11} + ... + 2hka^*b^* U_{12}]$.

Table 10. Hydrogen Coordinates [x 10^4] and Isotropic Displacement Parameters [$Å^2$ x 10^3] for **10**.

Atom	x	y	z	U(eq)
H(2A)	1411(2)	2139(2)	1959(1)	57
H(4A)	616(2)	4228(2)	169(2)	59
H(6A)	3069(1)	4386(2)	1720(1)	53
H(7A)	2714(2)	2370(2)	3084(1)	70
H(7B)	2983(2)	3463(2)	3180(1)	70
H(7C)	3230(2)	3486(2)	3077(1)	70
H(7D)	2582(2)	2628(2)	3181(1)	70
H(8AA)	3981(3)	1948(4)	3135(3)	74
H(8AB)	4241(3)	2812(4)	2579(3)	74
H(9AA)	3847(4)	1548(4)	1518(3)	72
H(9AB)	3065(4)	1326(4)	2047(3)	72
H(8BB)	4173(3)	2163(3)	3060(3)	69
H(8BA)	3361(3)	1490(3)	2698(3)	69
H(9BA)	4191(3)	2817(4)	1733(3)	69
H(9BB)	3989(3)	1729(4)	1501(3)	69
H(10A)	-150(2)	2307(2)	1391(2)	90
H(10B)	-475(2)	3264(2)	910(2)	90
H(10C)	-512(2)	3203(2)	651(2)	90
H(10D)	-222(2)	2526(2)	1461(2)	90
H(11A)	-538(3)	2608(4)	-370(4)	85
H(11B)	-864(3)	1756(4)	166(4)	85
H(12A)	647(3)	1167(4)	559(3)	74
H(12B)	533(3)	1340(4)	-449(3)	74
H(11C)	-56(4)	1206(5)	549(4)	106
H(11D)	-740(4)	1774(5)	-135(4)	106
H(12C)	764(4)	1336(5)	-467(4)	101
H(12D)	312(4)	2311(5)	-813(4)	101
H(13A)	1409(2)	5826(2)	664(2)	86
H(13B)	2454(2)	5817(2)	984(2)	86
H(13C)	1366(2)	5777(2)	443(2)	86
H(13D)	2273(2)	5851(2)	1104(2)	86
H(14A)	2339(2)	6178(2)	-490(2)	90

Table 10 (continued)

H(14B)	1576(2)	5405(2)	-751(2)	90
H(15A)	2933(3)	4592(4)	-857(3)	70
H(15B)	3366(3)	4943(4)	82(3)	70
H(14C)	3026(9)	5833(7)	57(7)	125
H(14D)	2112(9)	6045(7)	-551(7)	125
H(15C)	1983(5)	4568(5)	-1040(4)	104
H(15D)	3024(5)	4525(5)	-719(4)	104
H(16A)	865(2)	4719(2)	2653(2)	76