(1α,5α,6β,7α,8β,9β)-3-Ethynylhexahydrotrispiro[2,4,10-trioxaadamantane-6,2'(3'*H*);8,2''(3''*H*);9,2'''(3'''*H*)-trisfuran] (12). To a cooled (-78 °C) CH_2Cl_2 (15 mL) solution of oxalyl chloride (0.22 mL, 2.52 mmol) were added DMSO (0.35 mL, 4.93 mmol) in CH_2Cl_2 (1 mL) and **11** (423 mg, 1.24 mmol) in CH_2Cl_2 (5 mL) within 5 min. The mixture was stirred for 1 h at -78 °C, treated with triethylamine (0.87 mL, 6.21 mmol), warmed to rt for 1 h, and washed with deionized water. The aqueous layer was extracted with CH_2Cl_2 (3 x 20 mL) and the combined organic phases were dried over K_2CO_3 and concentrated in vacuo.

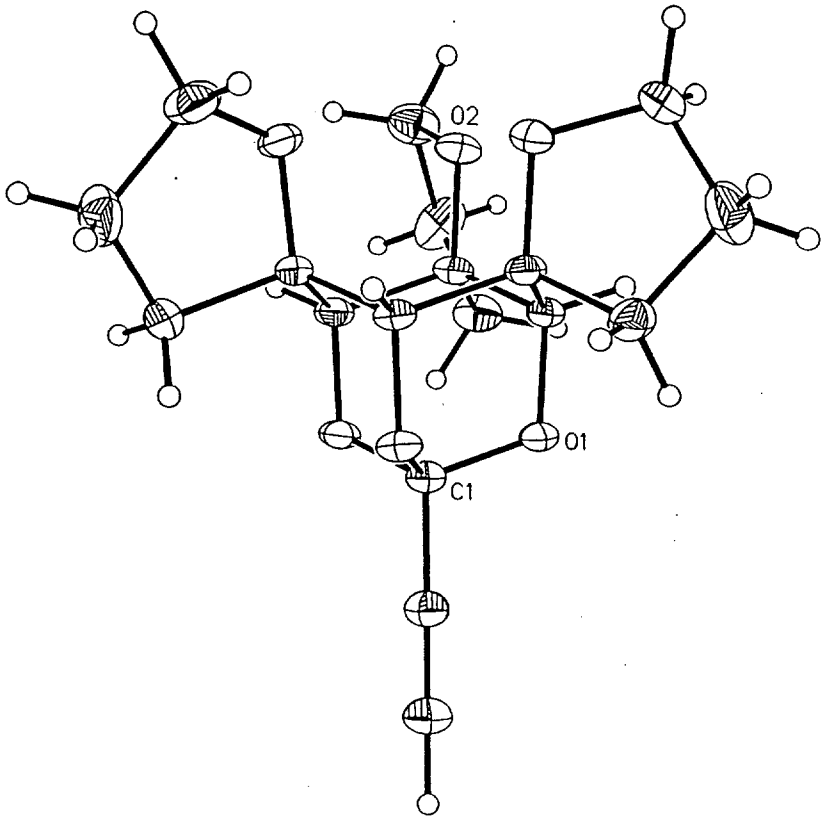
This residue was dissolved with K_2CO_3 (344 mg, 2.49 mmol) in methanol (15 mL) and treated with dimethyl 1-diazo-2-oxypropylphosphonate (286 mg, 1.49 mmol) for 6 h at rt. The reaction mixture was diluted with deionized water (20 mL) and CH_2Cl_2 (20 mL) and the separated aqueous layer was extracted with CH_2Cl_2 (3 x 20 mL). The combined organic phases were dried over K_2CO_3 and concentrated in vacuo. The solid residue was washed with 50% EtOAc-hexane and the remainder was dissolved in 10% MeOH-CH_2Cl_2. The solvent was removed in vacuo to give 135 mg (33% over 2 steps) of **12** as a white solid, mp >270 °C dec (from 5% MeOH in CH_2Cl_2); IR (film, cm^{-1}) 3400, 2100, 1615, 1255, 1040, 960; ^1H NMR (300 MHz, $CDCl_3$) δ 3.96 (t, J = 6.9 Hz, 6 H), 3.60 (s, 3 H), 2.53 (s, 1 H), 2.32 (t, J = 7.6 Hz, 6 H), 1.88 (m, 6 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 101.4, 79.2, 77.2, 77.0, 69.8, 69.3, 36.1, 23.6; HRMS *m/z* (M^+) calcd 334.1416, obsd 334.1430.

3,3''''-(Butadiynylene)bis[(1α,5α,6β,7α,8β,9β)-hexahydrotrispiro[2,4,10-trioxaadamantane-6,2'(3'*H*);8,2''(3''*H*);9,2'''(3'''*H*)-trisfuran]] (13). To a CH_2Cl_2 solution (15 mL) of **12** (128 mg, 0.383 mmol) and TMEDA (0.144 mL, 0.766 mmol) was added CuCl (38 mg, 0.38 mmol) under dry air conditions with protection by means of a $CaCl_2$ drying tube. After 4 h at rt, the reaction mixture was diluted with deionized water (20 mL), extracted with CH_2Cl_2 (4 x 30 mL), and dried over K_2CO_3. The concentrated filtrate was purified by chromatography on silica gel (elution with 5-10% MeOH in CH_2Cl_2) to afford 128 mg (100%) of the **13** as a white solid, mp >280 °C (from CH_2Cl_2); IR (film,

cm^{-1}) 3418, 1633, 1462, 1264; ^1H NMR (300 MHz, $CDCl_3$) δ 3.95 (t, J = 6.8 Hz, 12 H), 3.57 (s, 6 H), 2.25 (t, J = 7.6 Hz, 12 H), 1.87 (m, 12 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 102.3, 79.7, 77.7, 73.4, 69.8, 64.9, 36.6, 24.1; HRMS *m/z* (M^+) calcd 666.2676, obsd 666.2707.

Complexation of 13 to Lithium Tetrafluoroborate. To a CH_2Cl_2 solution (1.0 mL) of **13** (10 mg, 0.015 mmol) was added $LiBF_4$ (1.4 mg, 0.015 mmol) dissolved in CH_3CN (0.05 mL) dropwise. Solvent was allowed to evaporate during 12 h and the precipitate was triturated with CH_2Cl_2 (1.0 mL) and CH_3CN (1.0 mL). The solvent was decanted and the residue was dried under vacuo to give **4** (spacer = (C≡C-C≡C) as a powdery white solid (11 mg), mp >280 °C.

Complexation of 13 to Lithium Picrate. To a CH_2Cl_2 solution (0.5 mL) of **13** (9.0 mg, 0.014 mmol) was added lithium picrate (6.3 mg, 0.027 mmol) dissolved in CH_3CN (2.0 mL) dropwise. Yellow crystals of **15** suitable for X-ray diffraction analysis were deposited from the solution.



ORTEP diagram of **12**.

Table 1. Crystal data and structure refinement for **12**.

Compound	**12**
Color / Shape	colorless / fragment
Empirical formula	$C_{18}H_{22}O_6$
Formula weight	334.36
Temperature	173(2) K
Crystal system	Trigonal
Space group	$P\bar{3}$
Unit cell dimensions	a = 9.9051(3) Å $\alpha = 90^\circ$
(4108 reflections	b = 9.9051(3) Å $\beta = 90^\circ$
in full θ range)	c = 9.1592(4) Å $\gamma = 120^\circ$
Volume	778.23(5) $Å^3$
Z	2
Density (calculated)	1.427 Mg/m^3
Absorption coefficient	0.107 mm^{-1}
Diffractometer / scan	Siemens SMART / CCD area detector
Radiation / wavelength	MoKα (graphite monochrom.) / 0.71073 Å
F(000)	356
Crystal size	0.12 x 0.25 x 0.32 mm
θ range for data collection	2.22 to 27.85°
Index ranges	$-12 \le h \le 12$, $-11 \le k \le 12$, $-11 \le \ell \le 11$
Reflections collected	4905
Independent / observed refls.	1219(R_{int} = 0.0128) / 1151([I>2σ(I)])
Absorption correction	SADABS[1]
Range of relat. transm. factors	0.97 and 0.78
Refinement method	Full-matrix least-squares on F^2
Computing	SHELXTL, Ver. 5[2]
Data / restraints / parameters	1216 / 0 / 74
Goodness-of-fit on F^2	1.081
SHELX-93 weight parameters	0.0613, 0.3098
Final R indices [I>2σ(I)]	R1 = 0.0424, wR2 = 0.1169
R indices (all data)	R1 = 0.0442, wR2 = 0.1214
Extinction coefficient	0.009(8)
Largest diff. peak and hole	0.328 and -0.218 $eÅ^{-3}$

Table 2. Atomic Coordinates [x 10^4] and Equivalent Isotropic Displacement Parameters [$Å^2$ x 10^3] for **12**.

Atom	x/a	y/b	z/c	U(eq)
O(1)	5686(1)	3900(1)	9(1)	20(1)
O(2)	4774(1)	2201(1)	3669(1)	22(1)
C(1)	6667	3333	-462(2)	19(1)
C(2)	5640(1)	3953(1)	1596(1)	18(1)
C(3)	4938(1)	2264(1)	2119(1)	18(1)
C(4)	3270(1)	1203(2)	1540(1)	26(1)
C(5)	2454(2)	42(2)	2773(2)	36(1)
C(6)	3593(2)	643(2)	4044(1)	31(1)
C(7)	6667	3333	-2074(2)	23(1)
C(8)	6667	3333	-3367(2)	28(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 **12**.

O(1)-C(1)	1.4107(9)	O(1)-C(2)	1.4562(12)
O(2)-C(3)	1.4272(12)	O(2)-C(6)	1.436(2)
C(1)-C(7)	1.477(3)	C(2)-C(3)	1.533(2)
C(2)-C(3)#1	1.546(2)	C(3)-C(4)	1.542(2)
C(4)-C(5)	1.524(2)	C(5)-C(6)	1.520(2)
C(7)-C(8)	1.184(3)		

C(1)-O(1)-C(2)	111.10(10)	C(3)-O(2)-C(6)	107.38(8)
O(1)#2-C(1)-O(1)	111.09(7)	O(1)-C(1)-C(7)	107.80(8)
O(1)-C(2)-C(3)	106.12(8)	O(1)-C(2)-C(3)#1	107.00(8)
C(3)-C(2)-C(3)#1	114.71(10)	O(2)-C(3)-C(2)	109.75(9)
O(2)-C(3)-C(4)	104.61(9)	C(2)-C(3)-C(4)	112.54(9)
O(2)-C(3)-C(2)#2	112.24(9)	C(2)-C(3)-C(2)#2	106.79(10)
C(4)-C(3)-C(2)#2	111.00(9)	C(5)-C(4)-C(3)	104.05(10)
C(6)-C(5)-C(4)	105.23(10)	O(2)-C(6)-C(5)	106.47(10)
C(8)-C(7)-C(1)	180.0		

Symmetry transformations used to generate equivalent atoms:

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

Table 4. Anisotropic Displacement Parameters [$Å^2$ x 10^3] for **12**.

Atom	U11	U22	U33	U23	U13	U12
O(1)	25(1)	27(1)	12(1)	-1(1)	-2(1)	16(1)
O(2)	25(1)	25(1)	13(1)	1(1)	4(1)	10(1)
C(1)	22(1)	22(1)	13(1)	0	0	11(1)
C(2)	22(1)	23(1)	12(1)	-1(1)	-1(1)	13(1)
C(3)	19(1)	23(1)	12(1)	-1(1)	0(1)	9(1)
C(4)	19(1)	31(1)	22(1)	-2(1)	-2(1)	7(1)
C(5)	23(1)	32(1)	44(1)	13(1)	-2(1)	7(1)
C(6)	35(1)	28(1)	22(1)	5(1)	7(1)	9(1)
C(7)	27(1)	27(1)	16(1)	0	0	14(1)
C(8)	33(1)	33(1)	16(1)	0	0	16(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 **12**.

Atom	x	y	z	U(eq)
H(2A)	4952(1)	4374(1)	1903(1)	22
H(4A)	2747(1)	1813(2)	1337(1)	31
H(4B)	3282(1)	662(2)	636(1)	31
H(5A)	1465(2)	1(2)	3038(2)	43
H(5B)	2223(2)	-1015(2)	2483(2)	43
H(6A)	3053(2)	645(2)	4953(1)	37
H(6B)	4062(2)	-25(2)	4194(1)	37
H(8A)	6667	3333	-4437(2)	33

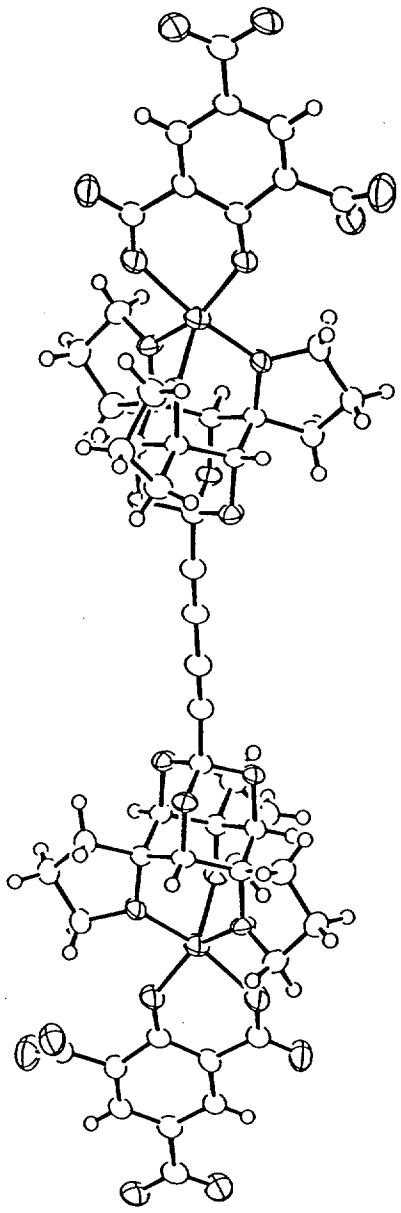


Figure 2. The crystallographically determined molecular structure of **15** drawn with 50% probability displacement ellipsoids for the non-hydrogen atoms. The hydrogen atoms are drawn with circles of arbitrary radii.

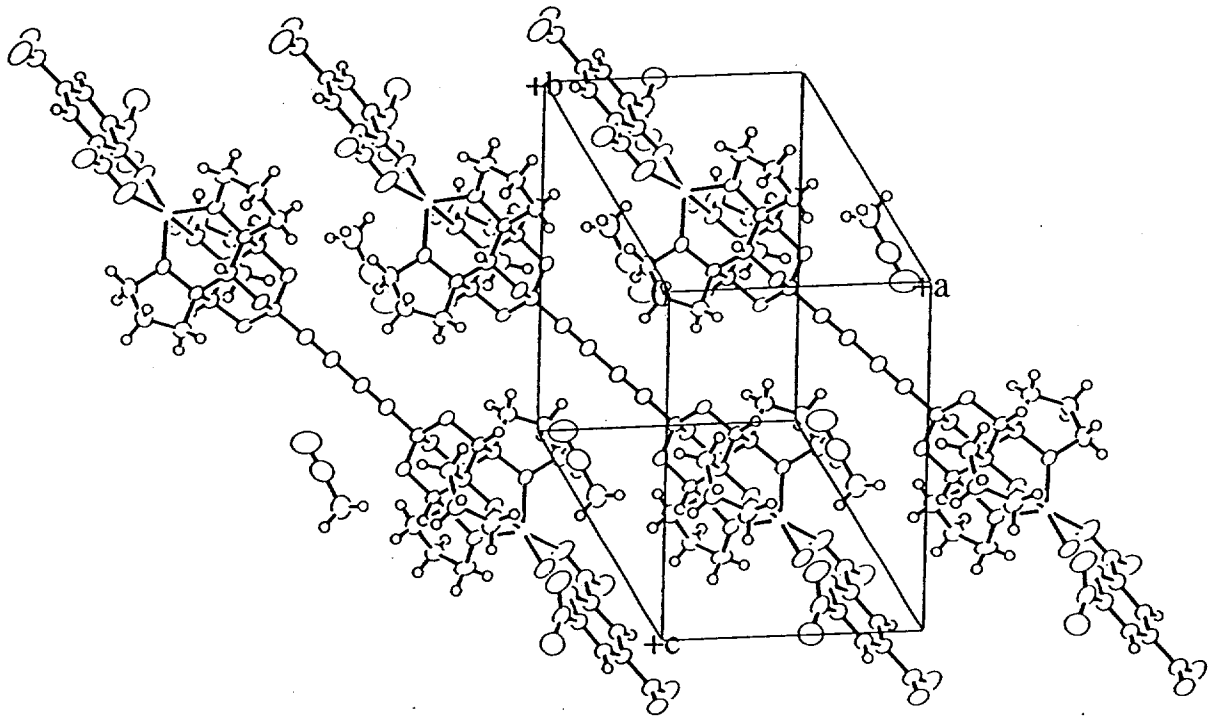


Figure 3. Packing diagram for **15**.

The data collection crystal was a clear yellow chunk which was mounted uncut. All work was done at 193 K using an Oxford Cryosystems Cryostream Cooler. Examination of the diffraction pattern on a Nonius Kappa CCD diffractometer indicated a triclinic crystal system. Almost a whole sphere of reciprocal space was measured using omega scans with a frame width of 1.0°. Data integration was done with Denzo[1], and scaling and merging of the data was done with Scalepack[1]. Merging the data and averaging the symmetry equivalent reflections (including the Friedel pairs) resulted in an Rint value of 0.042. The teXsan[2] package was used to determine the space group as $P\bar{1}$ based on the intensity statistics

The structure was solved by the direct methods procedure in SHELXS-86[3]. The molecule contains a crystallographic inversion center in the middle of the C(18)-C(18') bond. The Li^+ ion is bonded to three oxygen atoms of the organic moiety and two oxygen atoms of the picrate ion. The asymmetric unit also contains a molecule of CH_3CN. Full-matrix least-squares refinements based on F^2 were performed in SHELXL-93[4]. The C(14) atom is disordered over two sites: C(14) and C(14A). The occupancy factor of C(14) was refined and that of C(14A) was restricted accordingly.

The hydrogen atoms were included in the model at calculated positions using a riding model with U(H) = 1.2 * Ueq(attached atom). For the methyl group, the torsion angle which defines its orientation was allowed to refine, and these hydrogen atoms were assigned U(H) = 1.5 * Ueq(attached carbon atom). The final refinement cycle was based on all the 6170 intensities and 408 variables and resulted in agreement factors of R1(F) = 0.092 and wR2(F^2) = 0.154. For the subset of data with $I > 2\sigma(I)$, the R1(F) value is 0.055 for 4153 reflections. The final difference electron density map contains maximum and minimum peak heights of 0.28 and -0.28 e/$Å^3$. Neutral atom scattering factors were used and include terms for anomalous dispersion[5].

References

(1) DENZO: Otwinowski, Z. & Minor, W., Methods in Enzymology, Vol 276: Macromolecular Crystallography, part A, 307-326, (1997), Carter, Jr., C. W. & Sweet, R. M., Eds., Academic Press.

(2) teXsan: Crystal Structure Analysis Package, version 1.7-2, Molecular Structure Corporation, The Woodlands, TX (1995).

(3) SHELXS-86: Sheldrick, G. M., Acta Cryst., (1990), A46, 467-473.

(4) SHELXL-93: Sheldrick, G. M., Universitat Gottingen, Germany, 1993.

(5) International Tables for Crystallography (1992). Volume C. Dordrecht: Kluwer Academic Publishers.

Table 6. Crystallographic Details for **15**.

Empirical formula	C26 H26 Li N4 O13
Formula weight	609.45
Temperature	193 K
Wavelength	0.71073 A
Crystal system	triclinic
Space group	$P\bar{1}$
Unit cell dimensions	a = 9.1043(1) A alpha = 99.219(1) deg. b = 10.3872(2) A beta = 92.494(1) deg. c = 15.7522(3) A gamma = 112.508(1) deg.
Volume	1349.40(4) A^3
Z	2
Density (calculated)	1.500 Mg/m^3
Absorption coefficient	0.121 mm^-1
Crystal size	0.12 x 0.15 x 0.23 mm
Theta range for data collection	2.31 to 27.53 deg.
Index ranges	0<=h<=11, -13<=k<=12, -20<=l<=20
Reflections collected	30996
Independent reflections	6170 [R(int) = 0.042]
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6170 / 0 / 408
Goodness-of-fit on F^2	1.030
Final R indices [I>2sigma(I)]	R1 = 0.055, wR2 = 0.133
R indices (all data)	R1 = 0.092, wR2 = 0.154
Largest diff. peak and hole	0.28 and -0.28 e/A^3

Table 7. Bond lengths [A] and angles [deg] for 15.

Li-O(7)	1.900(4)
Li-O(3)	1.967(4)
Li-O(1)	2.002(4)
Li-O(2)	2.022(4)
Li-O(8)	2.108(4)
O(1)-C(1)	1.435(2)
O(1)-C(9)	1.435(2)
O(2)-C(12)	1.444(2)
O(2)-C(5)	1.447(2)
O(3)-C(15)	1.443(2)
O(3)-C(3)	1.444(2)
O(4)-C(16)	1.409(2)
O(4)-C(2)	1.453(2)
O(5)-C(16)	1.405(2)
O(5)-C(4)	1.455(2)
O(6)-C(16)	1.406(2)
O(6)-C(6)	1.455(2)
O(7)-C(19)	1.250(2)
O(8)-N(1)	1.235(2)
O(9)-N(1)	1.223(2)
O(10)-N(2)	1.223(2)
O(11)-N(2)	1.231(2)
O(12)-N(3)	1.222(3)
O(13)-N(3)	1.227(2)
N(1)-C(20)	1.448(3)
N(2)-C(22)	1.453(2)
N(3)-C(24)	1.464(3)
N(4)-C(25)	1.128(4)
C(1)-C(6)	1.527(3)
C(1)-C(7)	1.528(3)
C(1)-C(2)	1.538(3)
C(2)-C(3)	1.532(3)
C(2)-H(2)	0.98
C(3)-C(13)	1.532(3)
C(3)-C(4)	1.542(3)
C(4)-C(5)	1.530(3)
C(4)-H(4)	0.98
C(5)-C(6)	1.529(3)
C(5)-C(10)	1.543(3)
C(6)-H(6)	0.98
C(7)-C(8)	1.511(3)
C(7)-H(7A)	0.97
C(7)-H(7B)	0.97
C(8)-C(9)	1.503(3)
C(8)-H(8A)	0.97
C(8)-H(8B)	0.97
C(9)-H(9A)	0.97
C(9)-H(9B)	0.97
C(10)-C(11)	1.523(3)
C(10)-H(10A)	0.97
C(10)-H(10B)	0.97
C(11)-C(12)	1.506(3)
C(11)-H(11A)	0.97
C(11)-H(11B)	0.97
C(12)-H(12A)	0.97
C(12)-H(12B)	0.97
C(13)-C(14A)	1.506(13)
C(13)-C(14)	1.515(3)
C(13)-H(13A)	0.97
C(13)-H(13B)	0.97
C(13)-H(13C)	0.97
C(13)-H(13D)	0.97

C(14)-H(14A)	0.97
C(14)-H(14B)	0.97
C(14A)-C(15)	1.457(14)
C(14A)-H(14C)	0.97
C(14A)-H(14D)	0.97
C(15)-H(15A)	0.97
C(15)-H(15B)	0.97
C(15)-H(15C)	0.97
C(15)-H(15D)	0.97
C(16)-C(17)	1.471(3)
C(17)-C(18)	1.185(3)
C(18)-C(18)#1	1.380(4)
C(19)-C(20)	1.448(3)
C(19)-C(24)	1.450(3)
C(20)-C(21)	1.385(3)
C(21)-C(22)	1.380(3)
C(21)-H(21)	0.93
C(22)-C(23)	1.386(3)
C(23)-C(24)	1.361(3)
C(23)-H(23)	0.93
C(25)-C(26)	1.428(4)
C(26)-H(26A)	0.96
C(26)-H(26B)	0.96
C(26)-H(26C)	0.96
O(7)-Li-O(3)	124.1(2)
O(7)-Li-O(1)	93.5(2)
O(3)-Li-O(1)	89.3(2)
O(7)-Li-O(2)	148.0(2)
O(3)-Li-O(2)	87.91(14)
O(1)-Li-O(2)	87.3(2)
O(7)-Li-O(8)	82.05(14)
O(3)-Li-O(8)	103.0(2)
O(1)-Li-O(8)	167.4(2)
O(2)-Li-O(8)	90.3(2)
C(1)-O(1)-C(9)	110.07(14)
C(1)-O(1)-Li	122.18(14)
C(9)-O(1)-Li	125.2(2)
C(12)-O(2)-C(5)	109.08(14)
C(12)-O(2)-Li	128.3(2)
C(5)-O(2)-Li	121.8(2)
C(15)-O(3)-C(3)	110.26(14)
C(15)-O(3)-Li	126.8(2)
C(3)-O(3)-Li	122.5(2)
C(16)-O(4)-C(2)	110.55(13)
C(16)-O(5)-C(4)	110.80(13)
C(16)-O(6)-C(6)	110.80(14)
C(19)-O(7)-Li	136.2(2)
N(1)-O(8)-Li	135.4(2)
O(9)-N(1)-O(8)	121.4(2)
O(9)-N(1)-C(20)	118.8(2)
O(8)-N(1)-C(20)	119.9(2)
O(10)-N(2)-O(11)	123.2(2)
O(10)-N(2)-C(22)	118.7(2)
O(11)-N(2)-C(22)	118.1(2)
O(12)-N(3)-O(13)	123.7(2)
O(12)-N(3)-C(24)	118.3(2)
O(13)-N(3)-C(24)	118.0(2)
O(1)-C(1)-C(6)	109.9(2)
O(1)-C(1)-C(7)	105.24(14)
C(6)-C(1)-C(7)	112.2(2)
O(1)-C(1)-C(2)	110.1(2)
C(6)-C(1)-C(2)	107.3(2)
C(7)-C(1)-C(2)	112.1(2)
O(4)-C(2)-C(3)	106.15(14)

Table 7 (continued)

O(4)-C(2)-C(1)	106.52(14)
C(3)-C(2)-C(1)	115.1(2)
O(3)-C(3)-C(2)	110.28(14)
O(3)-C(3)-C(13)	105.1(2)
C(2)-C(3)-C(13)	112.6(2)
O(3)-C(3)-C(4)	110.36(14)
C(2)-C(3)-C(4)	106.6(2)
C(13)-C(3)-C(4)	112.0(2)
O(5)-C(4)-C(5)	106.34(14)
O(5)-C(4)-C(3)	106.26(14)
C(5)-C(4)-C(3)	115.1(2)
O(2)-C(5)-C(4)	109.4(2)
O(2)-C(5)-C(6)	110.3(2)
C(4)-C(5)-C(6)	107.2(2)
O(2)-C(5)-C(10)	106.0(2)
C(4)-C(5)-C(10)	112.3(2)
C(6)-C(5)-C(10)	111.6(2)
O(6)-C(6)-C(1)	105.75(14)
O(6)-C(6)-C(5)	106.46(14)
C(1)-C(6)-C(5)	115.2(2)
C(8)-C(7)-C(1)	103.4(2)
C(9)-C(8)-C(7)	103.3(2)
O(1)-C(9)-C(8)	107.5(2)
C(11)-C(10)-C(5)	104.0(2)
C(12)-C(11)-C(10)	102.2(2)
O(2)-C(12)-C(11)	105.0(2)
C(14A)-C(13)-C(3)	102.3(6)
C(14)-C(13)-C(3)	103.2(2)
C(13)-C(14)-C(15)	101.8(2)
C(15)-C(14A)-C(13)	105.2(8)
O(3)-C(15)-C(14A)	102.7(5)
O(3)-C(15)-C(14)	105.7(2)
O(5)-C(16)-O(6)	111.4(2)
O(5)-C(16)-O(4)	110.8(2)
O(6)-C(16)-O(4)	111.9(2)
O(5)-C(16)-C(17)	108.6(2)
O(6)-C(16)-C(17)	106.9(2)
O(4)-C(16)-C(17)	107.0(2)
C(18)-C(17)-C(16)	177.1(2)
C(17)-C(18)-C(18)#1	179.5(3)
O(7)-C(19)-C(20)	125.8(2)
O(7)-C(19)-C(24)	122.0(2)
C(20)-C(19)-C(24)	112.2(2)
C(21)-C(20)-N(1)	116.5(2)
C(21)-C(20)-C(19)	123.4(2)
N(1)-C(20)-C(19)	120.1(2)
C(22)-C(21)-C(20)	119.2(2)
C(21)-C(22)-C(23)	121.6(2)
C(21)-C(22)-N(2)	119.5(2)
C(23)-C(22)-N(2)	118.9(2)
C(24)-C(23)-C(22)	118.7(2)
C(23)-C(24)-C(19)	124.8(2)
C(23)-C(24)-N(3)	116.7(2)
C(19)-C(24)-N(3)	118.6(2)
N(4)-C(25)-C(26)	177.9(3)

Symmetry transformations used to generate equivalent atoms:
#1 -x,-y+1,-z+1

Table 8. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for **15**.

	x	y	z	U(eq)
Li	5155(4)	1346(4)	7523(2)	39(1)
O(1)	4942(2)	3186(1)	7934(1)	33(1)
O(2)	5534(2)	1938(2)	6363(1)	34(1)
O(3)	2853(2)	464(1)	7114(1)	32(1)
O(4)	1395(2)	3191(1)	6735(1)	32(1)
O(5)	1920(2)	2195(1)	5421(1)	32(1)
O(6)	3664(2)	4453(1)	6117(1)	31(1)
O(7)	6041(2)	1310(2)	8629(1)	44(1)
O(8)	5923(2)	-311(2)	7137(1)	56(1)
O(9)	6651(2)	-2047(2)	7119(1)	51(1)
O(10)	8757(2)	-2999(2)	9636(1)	59(1)
O(11)	9048(2)	-1655(2)	10880(1)	47(1)
O(12)	6698(2)	1895(2)	11140(1)	65(1)
O(13)	7517(2)	3159(2)	10160(1)	60(1)
N(1)	6469(2)	-1015(2)	7514(1)	35(1)
N(2)	8626(2)	-1991(2)	10095(1)	39(1)
N(3)	7102(2)	2033(2)	10419(1)	42(1)
N(4)	1093(4)	4174(3)	2528(2)	100(1)
C(1)	4001(2)	3692(2)	7430(1)	29(1)
C(2)	2266(2)	2587(2)	7229(1)	28(1)
C(3)	2057(2)	1168(2)	6666(1)	28(1)
C(4)	2813(2)	1514(2)	5830(1)	29(1)
C(5)	4566(2)	2557(2)	5967(1)	30(1)
C(6)	4663(2)	3908(2)	6566(1)	29(1)
C(7)	4115(3)	5083(2)	7986(1)	36(1)
C(8)	4337(3)	4865(2)	8900(1)	40(1)
C(9)	5247(3)	3919(3)	8818(1)	50(1)
C(10)	5238(3)	2872(3)	5107(1)	41(1)
C(11)	6293(3)	2036(2)	4967(1)	38(1)
C(12)	6880(2)	2065(2)	5881(1)	40(1)
C(13)	302(2)	122(2)	6476(2)	39(1)
C(14)	405(3)	-1310(3)	6413(2)	39(1)*
C(14A)	216(16)	-903(14)	7067(11)	45(4)**
C(15)	1780(3)	-983(2)	7104(1)	42(1)
C(16)	2065(2)	3464(2)	5960(1)	30(1)
C(17)	1148(2)	4109(2)	5516(1)	34(1)
C(18)	426(2)	4676(2)	5190(1)	35(1)
C(19)	6663(2)	571(2)	8931(1)	33(1)
C(20)	6914(2)	-617(2)	8441(1)	31(1)
C(21)	7552(2)	-1437(2)	8814(1)	33(1)
C(22)	7942(2)	-1145(2)	9700(1)	34(1)
C(23)	7744(2)	-29(2)	10224(1)	35(1)
C(24)	7174(2)	805(2)	9847(1)	34(1)
C(25)	818(3)	4868(3)	2119(2)	63(1)
C(26)	520(3)	5789(3)	1615(2)	65(1)

*Occupancy factor is 0.829(7).
**Occupancy factor is 0.171.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

Table 9. Anisotropic displacement parameters ($A^2 \times 10^3$) for **15**.

The anisotropic displacement factor exponent takes the form:
$-2\pi^2 [h^2 a^{*2} U11 + ... + 2 h k a^* b^* U12]$

	U11	U22	U33	U23	U13	U12
Li	45(2)	43(2)	36(2)	8(2)	-4(2)	24(2)
O(1)	44(1)	34(1)	25(1)	3(1)	-6(1)	22(1)
O(2)	35(1)	45(1)	34(1)	15(1)	6(1)	26(1)
O(3)	37(1)	27(1)	36(1)	10(1)	-2(1)	15(1)
O(4)	34(1)	36(1)	33(1)	11(1)	3(1)	21(1)
O(5)	40(1)	35(1)	27(1)	6(1)	-5(1)	21(1)
O(6)	33(1)	33(1)	33(1)	13(1)	0(1)	16(1)
O(7)	62(1)	43(1)	35(1)	5(1)	-9(1)	33(1)
O(8)	88(1)	62(1)	34(1)	5(1)	-10(1)	51(1)
O(9)	80(1)	47(1)	36(1)	2(1)	0(1)	37(1)
O(10)	81(1)	47(1)	59(1)	5(1)	-12(1)	40(1)
O(11)	54(1)	56(1)	40(1)	21(1)	-1(1)	27(1)
O(12)	88(1)	73(1)	43(1)	6(1)	17(1)	42(1)
O(13)	94(1)	47(1)	47(1)	3(1)	-7(1)	39(1)
N(1)	41(1)	35(1)	32(1)	8(1)	0(1)	17(1)
N(2)	41(1)	36(1)	43(1)	15(1)	-4(1)	16(1)
N(3)	48(1)	46(1)	36(1)	1(1)	-6(1)	28(1)
N(4)	108(2)	85(2)	116(3)	53(2)	19(2)	33(2)
C(1)	34(1)	29(1)	27(1)	7(1)	-2(1)	17(1)
C(2)	34(1)	32(1)	26(1)	11(1)	3(1)	19(1)
C(3)	30(1)	27(1)	30(1)	8(1)	-1(1)	14(1)
C(4)	37(1)	31(1)	25(1)	6(1)	-3(1)	19(1)
C(5)	34(1)	36(1)	29(1)	13(1)	4(1)	21(1)
C(6)	29(1)	29(1)	32(1)	10(1)	-1(1)	14(1)
C(7)	47(1)	31(1)	34(1)	3(1)	-2(1)	19(1)
C(8)	53(1)	39(1)	32(1)	2(1)	0(1)	24(1)
C(9)	69(2)	66(2)	27(1)	-6(1)	-9(1)	46(1)
C(10)	49(1)	50(1)	36(1)	18(1)	12(1)	28(1)
C(11)	39(1)	40(1)	37(1)	7(1)	10(1)	17(1)
C(12)	34(1)	50(1)	42(1)	7(1)	8(1)	23(1)
C(13)	32(1)	34(1)	48(1)	9(1)	-1(1)	11(1)
C(14)	38(2)	31(1)	43(2)	6(1)	-2(1)	11(1)
C(14A)	52(9)	30(7)	54(10)	18(6)	17(6)	13(6)
C(15)	53(1)	30(1)	40(1)	13(1)	-3(1)	13(1)
C(16)	31(1)	32(1)	29(1)	9(1)	0(1)	15(1)
C(17)	35(1)	35(1)	36(1)	10(1)	0(1)	17(1)
C(18)	35(1)	34(1)	39(1)	11(1)	-2(1)	16(1)
C(19)	36(1)	33(1)	33(1)	8(1)	-3(1)	16(1)
C(20)	33(1)	32(1)	28(1)	6(1)	-3(1)	11(1)
C(21)	34(1)	31(1)	34(1)	7(1)	0(1)	13(1)
C(22)	35(1)	34(1)	36(1)	13(1)	-1(1)	15(1)
C(23)	38(1)	40(1)	28(1)	10(1)	-1(1)	17(1)
C(24)	38(1)	35(1)	31(1)	6(1)	0(1)	18(1)
C(25)	64(2)	53(2)	67(2)	13(1)	15(1)	18(1)
C(26)	59(2)	74(2)	73(2)	19(2)	14(1)	37(2)

Table 10. Calculated hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for **15**.

	x	y	z	U(eq)
H(2)	1801(2)	2421(2)	7770(1)	34
H(4)	2700(2)	633(2)	5445(1)	35
H(6)	5772(2)	4608(2)	6668(1)	35
H(7A)	5019(3)	5883(2)	7869(1)	44
H(7B)	3144(3)	5240(2)	7882(1)	44
H(8A)	4945(3)	5760(2)	9289(1)	48
H(8B)	3313(3)	4409(2)	9110(1)	48
H(9A)	6383(3)	4481(3)	8972(1)	60
H(9B)	4894(3)	3243(3)	9201(1)	60
H(10A)	4380(3)	2553(3)	4636(1)	49
H(10B)	5859(3)	3880(3)	5150(1)	49
H(11A)	5682(3)	1071(2)	4660(1)	46
H(11B)	7174(3)	2492(2)	4648(1)	46
H(12A)	7200(2)	1280(2)	5905(1)	48
H(12B)	7786(2)	2948(2)	6110(1)	48
H(13A)	-308(2)	287(2)	6940(2)	46*
H(13B)	-187(2)	197(2)	5936(2)	46*
H(13C)	-415(2)	597(2)	6614(2)	46**
H(13D)	43(2)	-354(2)	5873(2)	46**
H(14A)	644(3)	-1637(3)	5844(2)	46*
H(14B)	-579(3)	-2018(3)	6539(2)	46*
H(14C)	4(16)	-559(14)	7639(11)	53**
H(14D)	-622(16)	-1829(14)	6837(11)	53**
H(15A)	1393(3)	-1080(2)	7664(1)	50*
H(15B)	2318(3)	-1622(2)	6964(1)	50*
H(15C)	2003(3)	-1297(2)	7624(1)	50**
H(15D)	1853(3)	-1620(2)	6601(1)	50**
H(21)	7716(2)	-2176(2)	8472(1)	39
H(23)	7996(2)	147(2)	10821(1)	41
H(26A)	-563(8)	5352(10)	1342(11)	97
H(26B)	1241(17)	5961(18)	1180(9)	97
H(26C)	683(24)	6673(9)	1985(3)	97

*Occupancy factor is 0.829
**Occupancy factor is 0.171