Highly Enantioselective 1,2-Addition of Lithium Acetylide-Ephedrate Complexes: Spectroscopic Evidence for Reaction Proceeding *via* a 2:2 Tetramer, and X-ray Characterization of Related Complexes

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Supporting Information

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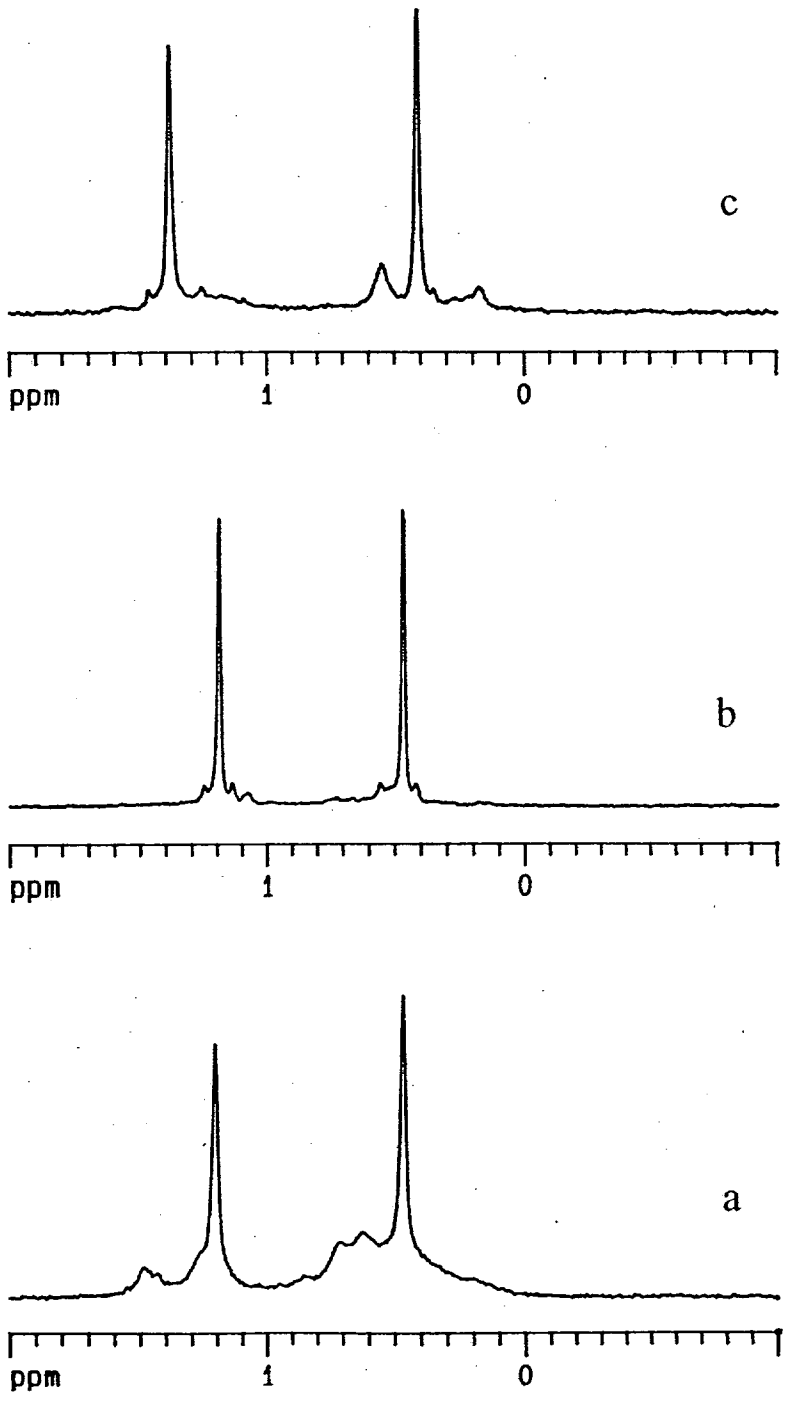
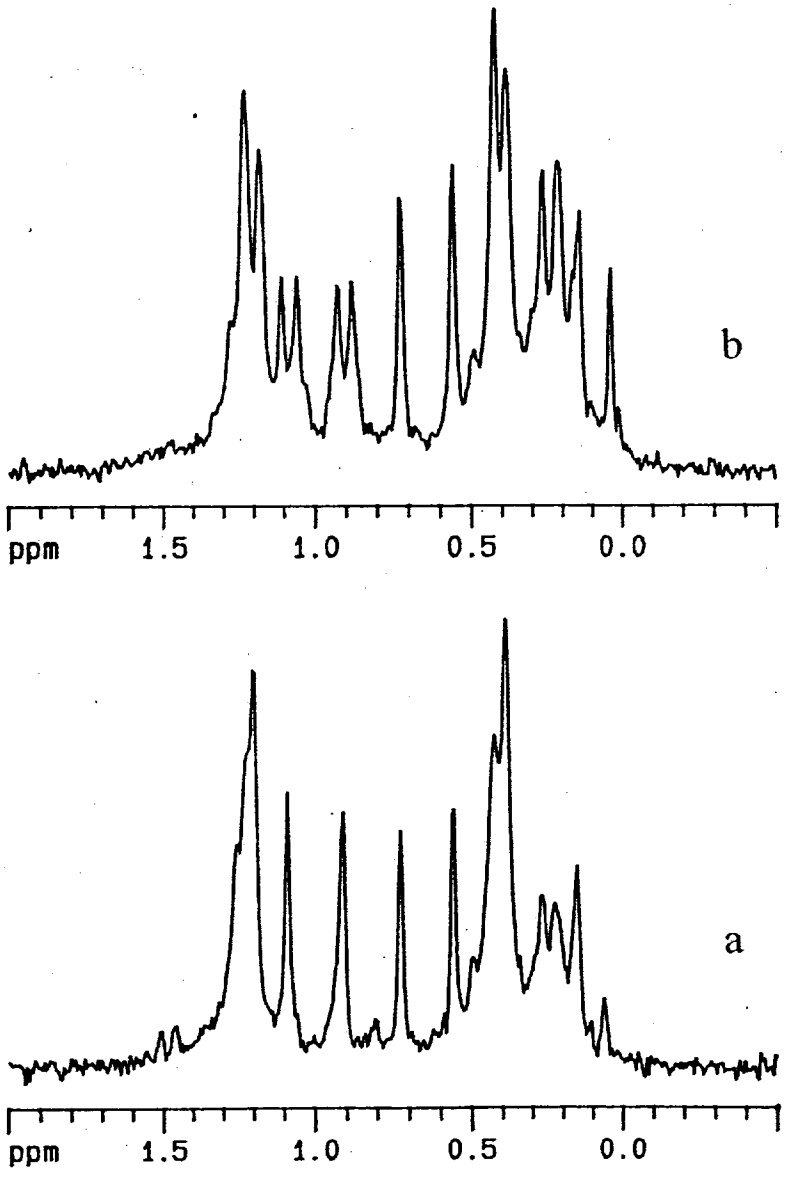
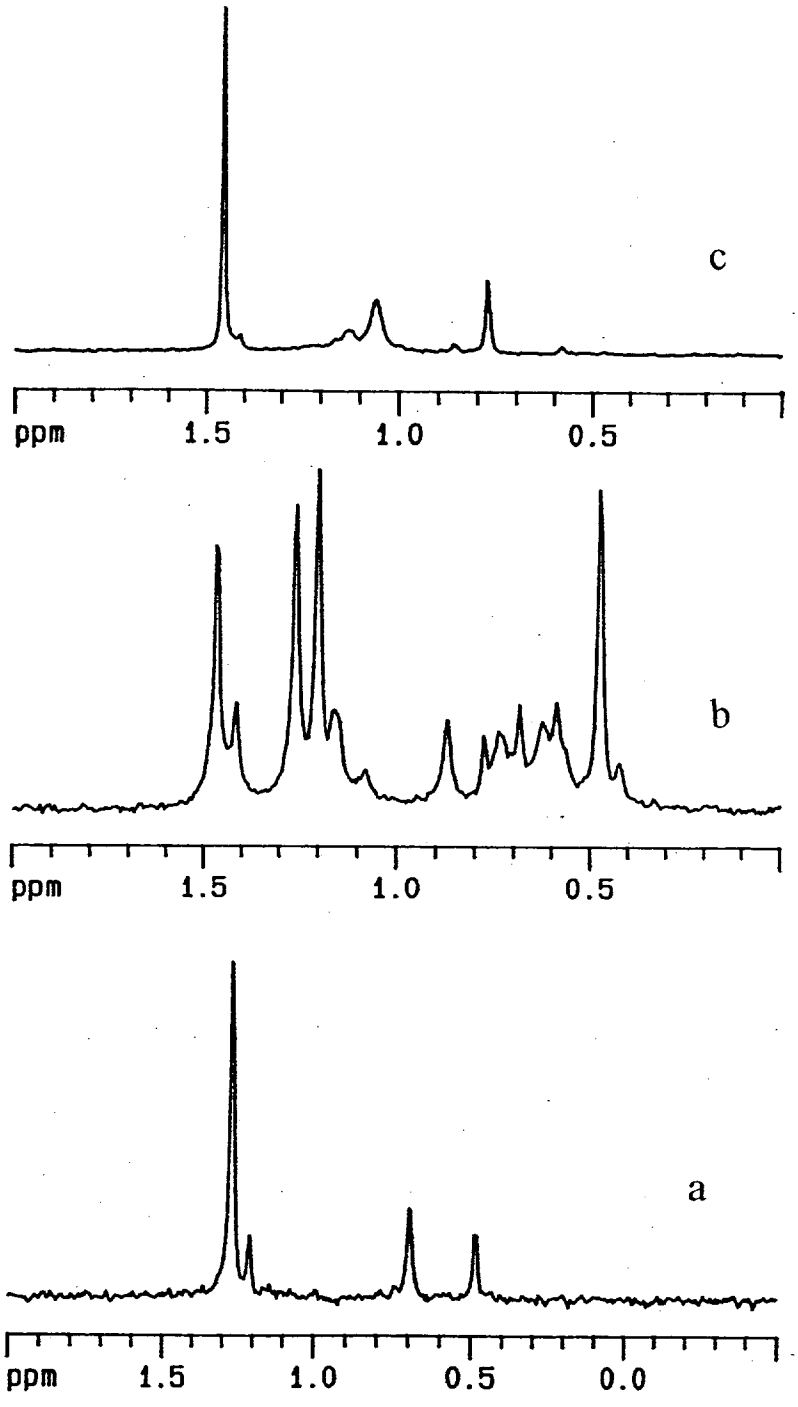


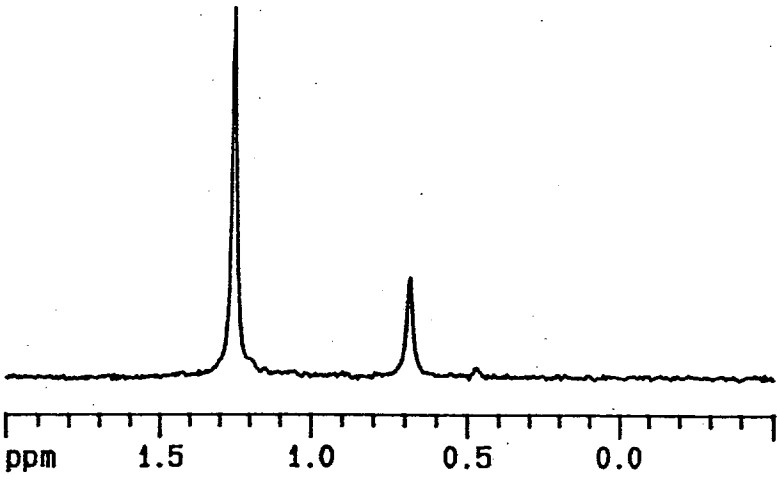
1. ^{6}Li NMR spectra for tetramers in Table 1: (a) **11** at –60°C; (b) **4** at –40°C; (c) **10** at –60°C



2. ^{6}Li NMR spectra after treatment of tetramer **6** with ketone **2** at –100°C: (a) using ^{15}N labeled ketone **2**; (b) using ^{15}N labeled ephedrine **4a**



3. ^{6}Li NMR spectra at –40°C: (a) 2:1 ratio of **4** and **5** generated in-situ and equilibrated at room temperature; (b) hexamer **12** crystals dissolved in THF-d8; (c) lithium ephedrate **4**



4. ^{6}Li NMR spectrum at –40°C of 3:1 tetramer

Supplemental Data for tetramer **8** (MSC Sample 97902)

Empirical Formula:	$C_{48}H_{67}Li_4N_3O_4$
Color of Crystal:	colorless
Crystal Dimensions were:	.35 x .35 x .38 mm.
Space Group:	$P2_12_12_1$

Cell Dimensions (at -165. C; 49 reflections measured at +/- 2-theta)

a =	11.022(4)
b =	17.118(6)
c =	24.372(9)

Z (Molecules/cell):	4
Volume:	4598.29
Calculated Density:	1.124
Wavelength:	.71069
Molecular Weight:	777.84
Linear Absorption Coefficient:	.644

A Picker four-circle goniostat equipped with a Furnas Monochromator (HOG crystal) driven by stepping motors (Slo-Syn) on each of the four axes. A locally designed ISA board in an IBM-PC compatible computer drives all motors. The computer also has a timer/scaler board that is used to accumulate the counts from the scintillation counter used with the goniostat.

Detector to sample distance =	22.5 cm.
Sample to source distance =	23.5 cm.
Take off angle =	2.0 deg.
Average omega scan width at half height =	.25 deg.
Scan speed =	8.0 deg/min
Scan width =	1.8 + dispersion
Single background time at extremes of scan =	4 sec.
Aperture size =	3.0 x 4.0 mm.
Limits of data collection were:	
Minimum two-theta =	6 deg.
Maximum two-theta =	45 deg.
Total number of reflections collected =	4201
Number of unique intensities =	3369
Number with F > 0.0 =	3017
Number with F > 2.33sigma(F) =	1908
R for Averaging =	.072
Final residuals are:	
R(F) =	.0726
Rw(F) =	.0500

Goodness of Fit for the last cycle =	1.535
Maximum delta/sigma for last cycle =	.12

Further details available on IUMSC data server at:
http://www.iumsc.indiana.edu/ Request data for structure 97902.

Fractional Coordinates and Isotropic Thermal Parameters for tetramer **8** (MSC Sample No. 97902)

Atom	x	y	z	Biso
LI(1)	2861(17)	387(11)	5630(7)	22(4)
LI(2)	3122(17)	324(11)	6692(7)	25
LI(3)	1975(17)	-742(12)	6197(9)	29
LI(4)	1094(17)	688(11)	6261(8)	26
C(5)	1086(11)	-65(7)	5495(5)	28
C(6)	173(13)	-354(7)	5392(6)	45
C(7)	-1657(13)	-627(12)	4747(6)	63
C(8)	-1081(17)	-724(11)	5289(7)	82
C(9)	-1164(21)	-1370(12)	4940(7)	100
C(10)	277(11)	379(8)	7804(5)	31
C(11)	-317(15)	637(7)	8277(5)	45
C(12)	-1286(16)	243(9)	8458(6)	53(4)
C(13)	-1799(12)	-338(8)	8161(6)	47
C(14)	-1208(12)	-593(6)	7686(5)	35
C(15)	-170(11)	-218(6)	7495(5)	22
C(16)	498(10)	-543(7)	6991(4)	18
O(17)	1419(7)	-55(4)	6796(3)	22
C(18)	1027(11)	-1369(6)	7119(4)	23
C(19)	2053(11)	-1314(6)	7530(5)	31
N(20)	1389(10)	-1723(5)	6589(4)	30
C(21)	2248(11)	-2367(7)	6659(5)	33
C(22)	2296(15)	-2723(8)	6089(7)	54
C(23)	1075(15)	-2527(8)	5834(5)	46
C(24)	390(14)	-2066(7)	6280(6)	49
C(25)	4409(10)	2290(6)	6528(5)	28
C(26)	5394(12)	2791(7)	6618(5)	31
C(27)	5546(13)	3451(8)	6291(5)	43
C(28)	4703(14)	3619(7)	5910(5)	41
C(29)	3703(12)	3137(7)	5817(5)	34
C(30)	3521(11)	2472(6)	6136(4)	19
C(31)	2418(11)	1937(7)	6047(4)	22
O(32)	2660(6)	1161(4)	6194(3)	18
C(33)	1359(10)	2269(6)	6375(4)	17
C(34)	1590(12)	2264(7)	6985(5)	36
N(35)	278(8)	1779(5)	6249(3)	17
C(36)	-1479(12)	1576(7)	5675(6)	41
C(37)	-1798(11)	1453(7)	6276(5)	39
C(38)	-802(11)	1918(6)	6582(4)	25
C(39)	-168(11)	1900(6)	5679(5)	28
C(40)	5349(11)	-1312(6)	6661(5)	24
C(41)	6141(11)	-1777(7)	6936(4)	25
C(42)	6881(11)	-2297(8)	6681(6)	34
C(43)	6763(11)	-2371(7)	6113(6)	36
C(44)	5945(11)	-1907(7)	5833(4)	25
C(45)	5211(10)	-1357(6)	6106(5)	19
C(46)	4351(10)	-841(6)	5787(5)	21
O(47)	3569(7)	-420(4)	6120(3)	21
C(48)	5022(9)	-291(6)	5394(4)	17
C(49)	5788(11)	304(6)	5703(5)	35
N(50)	4123(8)	115(5)	5031(3)	18

C(51)	3527(11)	-419(6)	4650(4)	25
C(52)	2899(10)	125(7)	4233(5)	33
C(53)	3701(13)	876(7)	4252(5)	41
C(54)	4687(11)	719(7)	4673(4)	32
O(55)	4237(7)	455(4)	7318(3)	27
C(56)	3819(13)	576(8)	7872(5)	43
C(57)	4964(14)	788(8)	8204(5)	44
C(58)	5815(12)	1103(7)	7763(6)	43
C(59)	5507(12)	591(7)	7290(5)	33
H(1)	-249*	-54*	471*	74
H(2)	-124*	-37*	445*	74
H(3)	-158*	-71*	561*	115
H(4)	-168*	-180*	504*	119
H(5)	-44*	-159*	477*	119
H(6)	103*	62*	770*	40
H(7)	-12*	110*	848*	56
H(8)	-163*	32*	880*	54
H(9)	-256*	-58*	826*	53
H(10)	-154*	-104*	748*	43
H(11)	-8*	-62*	671*	26
H(12)	43*	-167*	729*	36
H(13)	241*	-181*	760*	33
H(14)	270*	-97*	739*	33
H(15)	180*	-110*	787*	33
H(16)	303*	-219*	676*	41
H(17)	197*	-274*	692*	41
H(18)	292*	-251*	587*	55
H(19)	238*	-328*	610*	55
H(20)	121*	-221*	552*	46
H(21)	68*	-299*	574*	46
H(22)	-9*	-242*	650*	51
H(23)	-13*	-169*	612*	51
H(24)	433*	181*	672*	33
H(25)	596*	268*	689*	46
H(26)	621*	378*	636*	52
H(27)	485*	406*	568*	49
H(28)	314*	327*	554*	38
H(29)	221*	195*	566*	31
H(30)	118*	280*	626*	29
H(31)	182*	174*	710*	38
H(32)	224*	260*	709*	38
H(33)	89*	241*	720*	38
H(34)	-155*	108*	549*	48
H(35)	-205*	193*	551*	48
H(36)	-258*	168*	636*	50
H(37)	-180*	93*	638*	50
H(38)	-67*	172*	694*	39
H(39)	-97*	246*	660*	39
H(40)	-17*	243*	559*	38
H(41)	32*	162*	543*	38
H(42)	486*	-95*	686*	34
H(43)	621*	-172*	733*	36
H(44)	746*	-260*	688*	45
H(45)	723*	-275*	592*	40
H(46)	589*	-196*	544*	35
H(47)	389*	-117*	557*	29
H(48)	556*	-58*	517*	29

H(49)	620*	6*	601*	37
H(50)	637*	56*	549*	37
H(51)	527*	71*	587*	37
H(52)	408*	-74*	447*	37
H(53)	293*	-70*	484*	37
H(54)	291*	-10*	387*	42
H(55)	209*	24*	433*	42
H(56)	410*	97*	391*	49
H(57)	327*	132*	436*	49
H(58)	486*	118*	488*	39
H(59)	541*	53*	451*	39
H(60)	328*	102*	788*	49
H(61)	345*	14*	801*	49
H(62)	477*	117*	847*	47
H(63)	526*	33*	838*	47
H(64)	564*	162*	769*	54
H(65)	662*	103*	788*	54
H(66)	596*	11*	731*	41
H(67)	573*	85*	696*	41

Notes:
1) Fractional coordinates are X 10**4 for non-hydrogen atoms and X 10**3 for hydrogen atoms. Biso values are X 10.
2) Isotropic values for those atoms refined anisotropically are calculated using the formula given by W. C. Hamilton, Acta Cryst., 12,609 (1959)
3) Parameters marked by an asterisk (*) were not varied.

Anisotropic Thermal Parameters
for tetramer **8** (MSC Sample No. 97902) (Uij form).

Atom	U11	U22	U33	U12	U13	U23
LI(2)	38(14)	26(13)	33(12)	-12(10)	-3(11)	19(11)
LI(3)	20(12)	38(13)	53(15)	-5(10)	18(12)	-25(13)
LI(4)	29(14)	35(13)	34(13)	-7(11)	-4(11)	7(11)
C(5)	32(9)	28(8)	46(9)	-11(7)	2(7)	-3(7)
C(6)	27(10)	35(9)	107(14)	-6(8)	-35(10)	33(9)
C(7)	52(12)	124(17)	64(12)	7(11)	-35(9)	-50(12)
C(8)	97(17)	104(17)	111(16)	25(13)	-6(14)	5(14)
C(9)	193(24)	106(17)	80(14)	-26(17)	-68(15)	-63(13)
C(10)	44(9)	48(9)	27(8)	15(8)	-5(7)	13(7)
C(11)	91(13)	32(9)	48(10)	41(9)	-5(9)	-10(8)
C(13)	59(11)	53(11)	67(10)	36(9)	29(9)	43(9)
C(14)	50(10)	17(7)	67(10)	20(7)	7(9)	8(7)
C(15)	33(9)	19(7)	33(7)	11(6)	6(7)	12(6)
C(16)	18(8)	28(8)	23(7)	14(6)	-6(6)	0(6)
O(17)	26(5)	24(5)	34(5)	-7(4)	10(4)	4(4)
C(18)	41(9)	26(8)	22(7)	-17(7)	4(7)	-2(6)
C(19)	50(9)	24(7)	44(8)	6(7)	15(8)	4(7)
N(20)	65(8)	20(6)	27(7)	-5(6)	31(7)	-3(5)
C(21)	44(9)	21(8)	62(10)	-1(7)	27(8)	-11(8)
C(22)	93(13)	29(9)	84(12)	-4(9)	25(12)	-14(9)
C(23)	106(14)	38(9)	29(9)	-34(9)	10(10)	-11(7)
C(24)	99(13)	30(9)	57(10)	-49(9)	5(10)	-7(8)
C(25)	21(8)	20(7)	65(10)	-8(6)	-1(8)	11(7)
C(26)	42(9)	28(8)	47(9)	5(7)	-6(8)	14(7)
C(27)	52(11)	62(11)	49(10)	-11(9)	17(9)	-11(9)
C(28)	72(12)	37(9)	48(10)	-17(9)	-8(9)	7(8)

C(29)	51(10)	35(9)	42(8)	-18(8)	7(8)	3(8)
C(30)	35(8)	24(7)	14(7)	-7(7)	3(7)	-2(6)
C(31)	42(9)	32(8)	9(7)	7(7)	-4(7)	1(6)
O(32)	25(5)	16(5)	29(5)	2(4)	2(4)	-2(4)
C(33)	23(7)	18(7)	26(7)	2(6)	8(7)	2(6)
C(34)	62(10)	50(9)	26(8)	-2(8)	13(8)	-33(7)
N(35)	17(6)	26(6)	21(6)	-3(5)	6(5)	0(5)
C(36)	48(10)	43(9)	65(10)	-10(8)	-7(9)	-2(8)
C(37)	41(9)	45(9)	60(10)	-11(7)	5(8)	5(8)
C(38)	32(8)	31(8)	32(8)	1(6)	6(7)	5(6)
C(39)	47(10)	30(8)	30(8)	-13(7)	-4(7)	8(7)
C(40)	35(9)	16(7)	39(9)	6(6)	-15(8)	5(6)
C(41)	30(9)	36(8)	30(8)	0(7)	2(7)	3(7)
C(42)	30(9)	37(9)	60(10)	11(7)	-4(8)	23(8)
C(43)	43(10)	46(10)	49(10)	9(8)	-10(9)	1(8)
C(44)	45(9)	25(8)	25(7)	-6(7)	8(7)	3(7)
C(45)	21(8)	21(7)	28(8)	-11(6)	0(7)	-3(6)
C(46)	16(7)	28(7)	38(8)	1(6)	-4(7)	-10(7)
O(47)	35(5)	19(4)	24(4)	3(4)	4(4)	-8(4)
C(48)	13(7)	20(7)	32(7)	12(6)	4(6)	-4(6)
C(49)	45(9)	25(8)	61(9)	-25(7)	-11(8)	9(7)
N(50)	30(7)	19(6)	19(5)	-1(5)	2(5)	3(5)
C(51)	46(9)	27(7)	22(7)	13(7)	-14(7)	-2(6)
C(52)	30(8)	53(9)	44(8)	-9(8)	5(7)	14(8)
C(53)	64(10)	44(9)	47(9)	3(8)	-22(9)	14(8)
C(54)	52(10)	47(9)	25(7)	-1(8)	17(7)	8(7)
O(55)	31(6)	36(5)	34(5)	-3(4)	0(5)	0(4)
C(56)	71(11)	72(11)	18(8)	-18(9)	5(8)	-1(7)

C(57)	80(12)	56(10)	32(9)	18(9)	-14(9)	-4(8)
C(58)	60(11)	44(9)	60(10)	-4(8)	-24(10)	-11(8)
C(59)	37(10)	52(9)	37(8)	-5(8)	-5(8)	2(7)

Form of the anisotropic thermal parameter:
exp[-(h**2b11+...+2hkb12+...)]
All values are X 10**4.

Bond Distances for tetramer **8** (MSC Sample 97902)

A	B	Distance	A	B	Distance
O(17)	C(16)	1.398(11)	C(27)	C(28)	1.345(23)
O(17)	LI(2)	2.002(20)	C(28)	C(29)	1.395(14)
O(17)	LI(3)	1.971(23)	C(29)	C(30)	1.393(12)
O(17)	LI(4)	1.856(20)	C(30)	C(31)	1.537(12)
O(32)	C(31)	1.402(5)	C(31)	C(33)	1.524(15)
O(32)	LI(1)	1.921(20)	C(33)	C(34)	1.510(23)
O(32)	LI(2)	1.946(18)	C(36)	C(37)	1.520(25)
O(32)	LI(4)	1.914(18)	C(36)	C(39)	1.547(15)
O(47)	C(46)	1.387(13)	C(37)	C(38)	1.547(15)
O(47)	LI(1)	1.985(18)	C(40)	C(41)	1.358(16)
O(47)	LI(2)	1.950(21)	C(40)	C(45)	1.362(22)
O(47)	LI(3)	1.851(19)	C(41)	C(42)	1.358(14)
O(55)	C(56)	1.443(22)	C(42)	C(43)	1.396(28)
O(55)	C(59)	1.420(13)	C(43)	C(44)	1.382(17)
O(55)	LI(2)	1.973(25)	C(44)	C(45)	1.408(14)
N(20)	C(18)	1.481(18)	C(45)	C(46)	1.512(15)
N(20)	C(21)	1.463(11)	C(46)	C(48)	1.533(15)
N(20)	C(24)	1.458(15)	C(48)	C(49)	1.521(13)
N(20)	LI(3)	2.037(16)	C(51)	C(52)	1.542(18)
N(35)	C(33)	1.489(10)	C(52)	C(53)	1.562(10)
N(35)	C(38)	1.459(15)	C(53)	C(54)	1.519(22)
N(35)	C(39)	1.487(20)	C(56)	C(57)	1.542(21)
N(35)	LI(4)	2.073(9)	C(57)	C(58)	1.524(22)
N(50)	C(48)	1.500(15)	C(58)	C(59)	1.488(19)
N(50)	C(51)	1.459(15)			
N(50)	C(54)	1.489(14)			
N(50)	LI(1)	2.070(25)			
C(5)	C(6)	1.149(14)			
C(5)	LI(1)	2.130(21)			
C(5)	LI(3)	2.289(26)			
C(5)	LI(4)	2.269(26)			
C(6)	C(8)	1.541(20)			
C(7)	C(8)	1.48(4)			
C(7)	C(9)	1.461(16)			
C(8)	C(9)	1.399(23)			
C(10)	C(11)	1.397(21)			
C(10)	C(15)	1.361(14)			
C(11)	C(12)	1.338(19)			
C(12)	C(13)	1.353(17)			
C(13)	C(14)	1.399(21)			
C(14)	C(15)	1.391(15)			
C(15)	C(16)	1.537(18)			
C(16)	C(18)	1.560(7)			
C(18)	C(19)	1.513(18)			
C(21)	C(22)	1.518(23)			
C(22)	C(23)	1.520(19)			
C(23)	C(24)	1.542(22)			
C(25)	C(26)	1.401(12)			
C(25)	C(30)	1.403(19)			
C(26)	C(27)	1.392(14)			

Bond Angles for tetramer **8** (MSC Sample 97902)

A	B	C	Angle
C(16)	O(17)	LI(2)	156.5(7)
C(16)	O(17)	LI(3)	97.0(7)
C(16)	O(17)	LI(4)	120.6(9)
LI(2)	O(17)	LI(3)	78.9(8)
LI(2)	O(17)	LI(4)	82.5(8)
LI(3)	O(17)	LI(4)	87.1(9)
C(31)	O(32)	LI(1)	119.6(10)
C(31)	O(32)	LI(2)	155.1(10)
C(31)	O(32)	LI(4)	104.5(7)
LI(1)	O(32)	LI(2)	84.7(9)
LI(1)	O(32)	LI(4)	82.7(8)
LI(2)	O(32)	LI(4)	82.6(8)
C(46)	O(47)	LI(1)	104.7(9)
C(46)	O(47)	LI(2)	156.2(9)
C(46)	O(47)	LI(3)	119.7(8)
LI(1)	O(47)	LI(2)	82.9(9)
LI(1)	O(47)	LI(3)	84.0(8)
LI(2)	O(47)	LI(3)	83.2(9)
C(56)	O(55)	C(59)	109.7(12)
C(56)	O(55)	LI(2)	122.8(10)
C(59)	O(55)	LI(2)	126.6(12)
C(18)	N(20)	C(21)	112.5(11)
C(18)	N(20)	C(24)	114.3(11)
C(18)	N(20)	LI(3)	99.0(7)
C(21)	N(20)	C(24)	104.2(6)
C(21)	N(20)	LI(3)	118.1(8)
C(24)	N(20)	LI(3)	109.2(10)
C(33)	N(35)	C(38)	116.5(8)
C(33)	N(35)	C(39)	112.3(8)
C(33)	N(35)	LI(4)	99.0(6)
C(38)	N(35)	C(39)	103.1(9)
C(38)	N(35)	LI(4)	119.5(8)
C(39)	N(35)	LI(4)	106.4(9)
C(48)	N(50)	C(51)	112.5(5)
C(48)	N(50)	C(54)	113.0(8)
C(48)	N(50)	LI(1)	97.6(10)
C(51)	N(50)	C(54)	104.5(10)
C(51)	N(50)	LI(1)	106.7(9)
C(54)	N(50)	LI(1)	122.6(5)
C(6)	C(5)	LI(1)	174.1(13)
C(6)	C(5)	LI(3)	108.7(10)
C(6)	C(5)	LI(4)	115.4(14)
LI(1)	C(5)	LI(3)	71.0(8)
LI(1)	C(5)	LI(4)	70.3(9)
LI(3)	C(5)	LI(4)	70.7(7)
C(5)	C(6)	C(8)	176.3(20)
C(8)	C(7)	C(9)	56.9(11)
C(6)	C(8)	C(7)	119.0(16)
C(6)	C(8)	C(9)	118.8(18)
C(7)	C(8)	C(9)	61.0(14)
C(7)	C(9)	C(8)	62.1(14)
C(11)	C(10)	C(15)	121.6(11)
C(10)	C(11)	C(12)	119.1(11)
C(11)	C(12)	C(13)	121.9(18)

C(12)	C(13)	C(14)	118.5(13)
C(13)	C(14)	C(15)	121.0(10)
C(10)	C(15)	C(14)	117.4(12)
C(10)	C(15)	C(16)	122.7(10)
C(14)	C(15)	C(16)	119.6(8)
O(17)	C(16)	C(15)	113.8(6)
O(17)	C(16)	C(18)	109.7(8)
C(15)	C(16)	C(18)	110.4(10)
N(20)	C(18)	C(16)	107.4(10)
N(20)	C(18)	C(19)	113.6(10)
C(16)	C(18)	C(19)	110.8(6)
N(20)	C(21)	C(22)	102.7(11)
C(21)	C(22)	C(23)	104.7(11)
C(22)	C(23)	C(24)	105.0(14)
N(20)	C(24)	C(23)	101.5(12)
C(26)	C(25)	C(30)	120.7(8)
C(25)	C(26)	C(27)	120.1(13)
C(26)	C(27)	C(28)	119.0(10)
C(27)	C(28)	C(29)	122.2(9)
C(28)	C(29)	C(30)	120.4(12)
C(25)	C(30)	C(29)	117.5(9)
C(25)	C(30)	C(31)	121.0(7)
C(29)	C(30)	C(31)	121.5(11)
O(32)	C(31)	C(30)	112.2(8)
O(32)	C(31)	C(33)	111.4(9)
C(30)	C(31)	C(33)	108.1(8)
N(35)	C(33)	C(31)	107.2(7)
N(35)	C(33)	C(34)	109.6(10)
C(31)	C(33)	C(34)	112.7(11)
C(37)	C(36)	C(39)	105.0(12)
C(36)	C(37)	C(38)	103.2(10)
N(35)	C(38)	C(37)	103.1(11)
N(35)	C(39)	C(36)	105.4(11)
C(41)	C(40)	C(45)	121.9(10)
C(40)	C(41)	C(42)	122.9(15)
C(41)	C(42)	C(43)	117.2(12)
C(42)	C(43)	C(44)	119.9(10)
C(43)	C(44)	C(45)	121.7(14)
C(40)	C(45)	C(44)	116.3(10)
C(40)	C(45)	C(46)	123.2(9)
C(44)	C(45)	C(46)	120.5(13)
O(47)	C(46)	C(45)	113.1(12)
O(47)	C(46)	C(48)	110.3(4)
C(45)	C(46)	C(48)	112.2(9)
N(50)	C(48)	C(46)	109.5(8)
N(50)	C(48)	C(49)	110.4(4)
C(46)	C(48)	C(49)	111.7(11)
N(50)	C(51)	C(52)	104.1(5)
C(51)	C(52)	C(53)	102.9(9)
C(52)	C(53)	C(54)	106.3(7)
N(50)	C(54)	C(53)	102.7(9)
O(55)	C(56)	C(57)	105.2(12)
C(56)	C(57)	C(58)	102.5(14)
C(57)	C(58)	C(59)	101.4(9)
O(55)	C(59)	C(58)	106.5(12)
O(32)	LI(1)	O(47)	95.5(12)
O(32)	LI(1)	N(50)	137.5(8)

O(32)	LI(1)	C(5)	104.8(10)
O(47)	LI(1)	N(50)	90.2(6)
O(47)	LI(1)	C(5)	101.6(6)
N(50)	LI(1)	C(5)	115.2(11)
O(17)	LI(2)	O(32)	94.2(8)
O(17)	LI(2)	O(47)	96.6(7)
O(17)	LI(2)	O(55)	121.5(13)
O(32)	LI(2)	O(47)	95.8(12)
O(32)	LI(2)	O(55)	124.2(5)
O(47)	LI(2)	O(55)	118.0(9)
O(17)	LI(3)	O(47)	101.1(9)
O(17)	LI(3)	N(20)	92.7(11)
O(17)	LI(3)	C(5)	96.8(6)
O(47)	LI(3)	N(20)	126.5(9)
O(47)	LI(3)	C(5)	100.3(10)
N(20)	LI(3)	C(5)	129.2(9)
O(17)	LI(4)	O(32)	100.2(10)
O(17)	LI(4)	N(35)	135.4(14)
O(17)	LI(4)	C(5)	100.9(4)
O(32)	LI(4)	N(35)	90.5(3)
O(32)	LI(4)	C(5)	100.0(11)
N(35)	LI(4)	C(5)	119.8(11)

Supplemental data for hexamer **12** (MSC Sample 97903)

Empirical Formula:	$C_{62}H_{80}Li_6N_4O_4$
Color of Crystal:	colorless
Crystal Dimensions were:	.30 x .30 x .42 mm.
Space Group:	$C222_1$

Cell Dimensions (at -174. C;171 reflections measured at +/- 2-theta)

a =	12.776(1)
b =	22.498(2)
c =	20.604(1)

Z (Molecules/cell):	4
Volume:	5922.33
Calculated Density:	1.107
Wavelength:	.71069
Molecular Weight:	986.98
Linear Absorption Coefficient:	.622

A Picker four-circle goniostat equipped with a Furnas Monochromator (HOG crystal) driven by stepping motors (Slo-Syn) on each of the four axes. A locally designed ISA board in an IBM-PC compatible computer drives all motors. The computer also has a timer/scaler board that is used to accumulate the counts from the scintillation counter used with the goniostat.

Detector to sample distance =	22.5 cm.
Sample to source distance =	23.5 cm.
Take off angle =	2.0 deg.
Average omega scan width at half height =	.25 deg.
Scan speed =	4.0 deg/min
Scan width =	2.0 + dispersion
Single background time at extremes of scan =	4 sec.
Aperture size =	3.0 x 4.0 mm.
Limits of data collection were:	
Minimum two-theta =	6 deg.
Maximum two-theta =	45 deg.
Total number of reflections collected =	2824
Number of unique intensities =	2154
Number with F > 0.0 =	2095
Number with F > 2.33sigma(F) =	1875
R for Averaging =	.057
Final residuals are:	
R(F) =	.0643
Rw(F) =	.0630

Goodness of Fit for the last cycle =	2.638
Maximum delta/sigma for last cycle =	.05

Further details available on IUMSC data server at:
http://www.iumsc.indiana.edu/ Request data for structure 97903.

Fractional Coordinates and Isotropic Thermal Parameters for hexamer **12** (MSC Sample No. 97903)

Atom	x	y	z	Biso
LI(1)	734(8)	3844(4)	3009(5)	30
LI(2)	685(7)	2447(4)	2924(4)	23
LI(3)	1581(7)	3120(4)	2196(5)	26
C(4)	984(4)	3966(3)	1931(3)	28
C(5)	1064(6)	4343(4)	1552(5)	66
C(6)	1164(10)	4806(10)	1084(12)	232
C(7)	1054(12)	5308(7)	995(13)	225
C(8)	2128(8)	5123(6)	944(9)	147
C(9)	1850(7)	2208(3)	4078(3)	47
C(10)	2098(10)	1693(3)	4424(4)	62
C(11)	3073(12)	1618(4)	4633(5)	74
C(12)	3853(7)	2037(5)	4530(4)	67
C(13)	3588(6)	2574(3)	4176(3)	47
C(14)	2579(5)	2651(3)	3942(3)	28
C(15)	2295(4)	3198(3)	3553(3)	23
O(16)	1462(3)	3113(2)	3134(2)	22
C(17)	2080(4)	3730(2)	4018(3)	26
C(18)	1204(5)	3598(3)	4482(3)	38
N(19)	1860(4)	4251(2)	3621(3)	32
C(20)	2749(5)	4445(3)	3209(3)	37
C(21)	2544(6)	5074(3)	3022(4)	52
C(22)	1720(7)	5283(3)	3497(4)	52
C(23)	1654(6)	4795(3)	4000(4)	49
C(24)	175(5)	1223(3)	1606(3)	33
C(25)	-192(6)	670(3)	1402(3)	43
C(26)	127(6)	446(3)	817(3)	37
C(27)	817(5)	758(3)	441(3)	43
C(28)	1196(5)	1317(3)	666(3)	34
C(29)	898(5)	1550(2)	1248(3)	25
C(30)	1312(4)	2144(3)	1485(3)	26
O(31)	770(3)	2376(2)	2010(2)	22
C(32)	2479(5)	2113(3)	1642(3)	29
C(33)	2693(5)	1764(3)	2249(3)	34
N(34)	2872(4)	2727(2)	1699(2)	32
C(35)	2935(5)	3015(3)	1058(3)	42
C(36)	3691(6)	3567(4)	1178(5)	62
C(37)	4255(5)	3407(3)	1804(4)	49
C(38)	3949(5)	2775(3)	1953(4)	41
H(1)	71*	541*	60*	199
H(2)	89*	554*	135*	199
H(3)	263*	521*	127*	156
H(4)	248*	508*	54*	156
H(5)	115*	224*	392*	77
H(6)	158*	139*	448*	95
H(7)	322*	126*	485*	105
H(8)	455*	197*	468*	97
H(9)	410*	288*	412*	76
H(10)	290*	330*	331*	51
H(11)	269*	380*	427*	58
H(12)	115*	391*	479*	70
H(13)	57*	356*	425*	70
H(14)	134*	324*	470*	70

H(15)	339*	442*	344*	68
H(16)	279*	421*	283*	68
H(17)	317*	530*	308*	85
H(18)	227*	509*	259*	85
H(19)	192*	564*	371*	81
H(20)	105*	533*	330*	81
H(21)	98*	478*	420*	82
H(22)	219*	485*	432*	82
H(23)	-9*	140*	199*	64
H(24)	-66*	45*	167*	73
H(25)	-13*	7*	68*	68
H(26)	106*	61*	4*	71
H(27)	168*	154*	41*	64
H(28)	123*	241*	113*	55
H(29)	282*	192*	129*	60
H(30)	253*	200*	262*	63
H(31)	227*	142*	226*	63
H(32)	341*	166*	227*	63
H(33)	320*	277*	73*	74
H(34)	228*	317*	93*	74
H(35)	417*	358*	83*	92
H(36)	330*	392*	122*	92
H(37)	498*	344*	172*	80
H(38)	404*	368*	213*	80
H(39)	397*	271*	241*	73
H(40)	440*	249*	175*	73

Notes:
1) Fractional coordinates are X 10**4 for non-hydrogen atoms and X 10**3 for hydrogen atoms. Biso values are X 10.
2) Isotropic values for those atoms refined anisotropically are calculated using the formula given by W. C. Hamilton, Acta Cryst., 12,609 (1959)
3) Parameters marked by an asterisk (*) were not varied.

Anisotropic Thermal Parameters
for hexamer **12** (MSC Sample No. 97903) (Uij form).

LI(1)	39(6)	41(6)	35(6)	-4(5)	-14(5)	5(5)
LI(2)	23(5)	37(6)	26(5)	-6(4)	1(4)	4(4)
LI(3)	28(5)	40(6)	30(5)	2(4)	-1(4)	8(5)
C(4)	30(3)	38(4)	37(4)	-7(3)	1(3)	1(3)
C(5)	54(5)	90(7)	105(8)	1(5)	3(5)	62(7)
C(6)	69(8)	364(27)	450(34)	48(13)	39(13)	350(28)
C(7)	98(10)	175(15)	582(40)	28(9)	81(15)	278(22)
C(8)	73(7)	177(12)	308(21)	-40(8)	-4(10)	175(14)
C(9)	97(6)	54(5)	28(4)	-11(4)	-18(4)	3(4)
C(10)	153(9)	35(4)	46(5)	8(5)	-24(6)	1(4)
C(11)	178(12)	53(6)	52(6)	47(7)	17(7)	7(4)
C(12)	96(7)	128(8)	32(4)	72(7)	-2(4)	14(5)
C(13)	70(5)	74(5)	37(4)	28(4)	-5(4)	4(4)
C(14)	41(3)	38(4)	28(3)	11(3)	-3(3)	-3(3)
C(15)	22(3)	41(3)	23(3)	-3(3)	-6(3)	3(3)
O(16)	27(2)	32(2)	24(2)	-4(2)	0(2)	-3(2)
C(17)	41(4)	34(3)	24(3)	-2(3)	-11(3)	1(3)
C(18)	53(4)	51(4)	40(4)	13(3)	3(3)	-7(3)
N(19)	49(3)	30(3)	44(3)	-2(3)	-6(3)	-4(3)
C(20)	46(4)	44(4)	51(4)	-14(3)	-10(3)	9(3)
C(21)	60(4)	54(4)	82(6)	-12(4)	-1(5)	18(4)
C(22)	91(6)	34(4)	74(5)	-4(4)	-6(5)	-6(4)
C(23)	84(6)	49(5)	55(5)	4(4)	-14(4)	-9(4)
C(24)	49(4)	43(4)	35(4)	-6(3)	5(3)	-15(3)
C(25)	70(5)	52(4)	42(4)	-14(4)	6(4)	-1(4)
C(26)	55(4)	40(4)	44(4)	2(3)	2(4)	-14(3)
C(27)	51(4)	74(5)	40(4)	0(4)	4(4)	-35(4)

C(28)	46(4)	41(4)	42(4)	-4(3)	9(3)	-11(3)
C(29)	32(3)	37(3)	26(3)	12(3)	-5(3)	-3(3)
C(30)	36(3)	34(3)	27(3)	8(3)	1(3)	4(3)
O(31)	26(2)	35(2)	25(2)	3(2)	1(2)	-10(2)
C(32)	37(4)	40(4)	34(3)	4(3)	2(3)	-9(3)
C(33)	33(4)	55(4)	41(4)	8(3)	-8(3)	-3(3)
N(34)	32(3)	50(3)	37(3)	-2(3)	2(2)	-2(3)
C(35)	49(4)	66(5)	45(4)	0(4)	12(4)	8(4)
C(36)	50(5)	78(6)	108(7)	-16(4)	18(5)	17(6)
C(37)	46(4)	73(6)	68(5)	-16(4)	8(4)	-16(4)
C(38)	28(3)	70(5)	59(4)	-5(3)	3(3)	-15(4)

Form of the anisotropic thermal parameter:
exp[-(h**2b11+...+2hkb12+...)]
All values are X 10**4.

Bond Distances for hexamer **12** (MSC Sample 97903)

A	B	Distance
O(16)	C(15)	1.384(6)
O(16)	LI(1)	1.906(5)
O(16)	LI(2)	1.850(5)
O(16)	LI(3)	1.940(10)
O(31)	C(30)	1.387(5)
O(31)	LI(2)	1.870(9)
O(31)	LI(2)	1.892(10)
O(31)	LI(3)	2.005(5)
N(19)	C(17)	1.456(4)
N(19)	C(20)	1.483(8)
N(19)	C(23)	1.475(4)
N(19)	LI(1)	2.121(10)
N(34)	C(32)	1.4760(26)
N(34)	C(35)	1.474(7)
N(34)	C(38)	1.475(7)
N(34)	LI(3)	2.133(9)
C(4)	C(5)	1.157(7)
C(4)	LI(1)	2.215(11)
C(4)	LI(1)	2.262(11)
C(4)	LI(3)	2.121(5)
C(5)	C(6)	1.426(15)
C(6)	C(7)	1.152(5)
C(6)	C(8)	1.452(14)
C(7)	C(8)	1.438(16)
C(9)	C(10)	1.397(6)
C(9)	C(14)	1.392(7)
C(10)	C(11)	1.329(15)
C(11)	C(12)	1.387(11)
C(12)	C(13)	1.451(8)
C(13)	C(14)	1.388(9)
C(14)	C(15)	1.514(5)
C(15)	C(17)	1.558(5)
C(17)	C(18)	1.502(8)
C(20)	C(21)	1.4905(27)
C(21)	C(22)	1.512(10)
C(22)	C(23)	1.513(6)
C(24)	C(25)	1.395(4)
C(24)	C(29)	1.393(7)
C(25)	C(26)	1.368(8)
C(26)	C(27)	1.367(8)
C(27)	C(28)	1.425(4)
C(28)	C(29)	1.363(7)
C(29)	C(30)	1.517(4)
C(30)	C(32)	1.528(8)
C(32)	C(33)	1.501(6)
C(35)	C(36)	1.593(6)
C(36)	C(37)	1.520(10)
C(37)	C(38)	1.507(3)
LI(2)	LI(2)	2.473(16)
LI(2)	LI(3)	2.420(9)

Bond Angles for hexamer **12** (MSC Sample 97903)

A	B	C	Angle
C(15)	O(16)	LI(1)	109.91(26)
C(15)	O(16)	LI(2)	132.14(25)
C(15)	O(16)	LI(3)	124.1(4)
LI(1)	O(16)	LI(2)	113.9(4)
LI(1)	O(16)	LI(3)	84.1(3)
LI(2)	O(16)	LI(3)	79.3(3)
C(30)	O(31)	LI(2)	125.8(4)
C(30)	O(31)	LI(2)	146.7(4)
C(30)	O(31)	LI(3)	101.8(3)
LI(2)	O(31)	LI(2)	82.2(4)
LI(2)	O(31)	LI(3)	115.43(24)
LI(2)	O(31)	LI(3)	76.7(3)
C(17)	N(19)	C(20)	114.2(4)
C(17)	N(19)	C(23)	113.9(4)
C(17)	N(19)	LI(1)	96.71(24)
C(20)	N(19)	C(23)	101.3(3)
C(20)	N(19)	LI(1)	107.8(4)
C(23)	N(19)	LI(1)	123.5(5)
C(32)	N(34)	C(35)	111.0(4)
C(32)	N(34)	C(38)	114.41(27)
C(32)	N(34)	LI(3)	99.4(3)
C(35)	N(34)	C(38)	103.6(4)
C(35)	N(34)	LI(3)	106.9(3)
C(38)	N(34)	LI(3)	121.4(4)
C(5)	C(4)	LI(1)	102.5(5)
C(5)	C(4)	LI(1)	139.8(4)
C(5)	C(4)	LI(3)	143.1(6)
LI(1)	C(4)	LI(1)	77.9(4)
LI(1)	C(4)	LI(3)	103.37(26)
LI(1)	C(4)	LI(3)	71.9(3)
C(4)	C(5)	C(6)	179.8(8)
C(5)	C(6)	C(7)	144.4(15)
C(5)	C(6)	C(8)	124.7(13)
C(7)	C(6)	C(8)	65.9(8)
C(6)	C(7)	C(8)	67.1(9)
C(6)	C(8)	C(7)	47.0(4)
C(10)	C(9)	C(14)	123.0(8)
C(9)	C(10)	C(11)	118.8(7)
C(10)	C(11)	C(12)	122.6(5)
C(11)	C(12)	C(13)	118.3(8)
C(12)	C(13)	C(14)	119.7(5)
C(9)	C(14)	C(13)	117.5(4)
C(9)	C(14)	C(15)	121.9(5)
C(13)	C(14)	C(15)	120.5(4)
O(16)	C(15)	C(14)	113.70(26)
O(16)	C(15)	C(17)	110.7(3)
C(14)	C(15)	C(17)	110.0(4)
N(19)	C(17)	C(15)	107.9(4)
N(19)	C(17)	C(18)	111.9(4)
C(15)	C(17)	C(18)	111.80(26)
N(19)	C(20)	C(21)	106.9(5)
C(20)	C(21)	C(22)	104.6(5)
C(21)	C(22)	C(23)	104.8(4)
N(19)	C(23)	C(22)	103.3(4)

C(25)	C(24)	C(29)	122.3(5)
C(24)	C(25)	C(26)	119.6(5)
C(25)	C(26)	C(27)	120.2(3)
C(26)	C(27)	C(28)	119.2(5)
C(27)	C(28)	C(29)	122.1(5)
C(24)	C(29)	C(28)	116.6(3)
C(24)	C(29)	C(30)	121.7(4)
C(28)	C(29)	C(30)	121.7(4)
O(31)	C(30)	C(29)	114.2(4)
O(31)	C(30)	C(32)	109.9(4)
C(29)	C(30)	C(32)	111.5(3)
N(34)	C(32)	C(30)	107.80(26)
N(34)	C(32)	C(33)	111.2(4)
C(30)	C(32)	C(33)	112.2(4)
N(34)	C(35)	C(36)	103.7(5)
C(35)	C(36)	C(37)	103.6(3)
C(36)	C(37)	C(38)	105.8(4)
N(34)	C(38)	C(37)	103.8(4)
O(16)	LI(1)	N(19)	87.8(4)
O(16)	LI(1)	C(4)	125.6(3)
O(16)	LI(1)	C(4)	99.7(4)
N(19)	LI(1)	C(4)	125.9(4)
N(19)	LI(1)	C(4)	115.8(4)
C(4)	LI(1)	C(4)	100.4(4)
O(16)	LI(2)	O(31)	125.8(3)
O(16)	LI(2)	O(31)	105.7(4)
O(16)	LI(2)	LI(2)	123.04(27)
O(16)	LI(2)	LI(3)	51.97(27)
O(31)	LI(2)	O(31)	97.0(4)
O(31)	LI(2)	LI(2)	49.3(3)
O(31)	LI(2)	LI(2)	48.5(3)
O(31)	LI(2)	LI(3)	124.6(4)
O(31)	LI(2)	LI(3)	53.74(26)
LI(2)	LI(2)	LI(3)	84.1(4)
O(16)	LI(3)	O(31)	98.2(3)
O(16)	LI(3)	N(34)	122.4(4)
O(16)	LI(3)	C(4)	103.6(3)
O(16)	LI(3)	LI(2)	48.68(24)
O(31)	LI(3)	N(34)	87.86(21)
O(31)	LI(3)	C(4)	120.9(4)
O(31)	LI(3)	LI(2)	49.53(24)
N(34)	LI(3)	C(4)	121.8(4)
N(34)	LI(3)	LI(2)	113.80(19)
C(4)	LI(3)	LI(2)	123.4(4)