Synthesis of New Tren-based Tris-macrocycles as Potential Receptors for Anions. Anion Cluster Assembling inside the Cavity Generated by a Bowl-shaped Receptor

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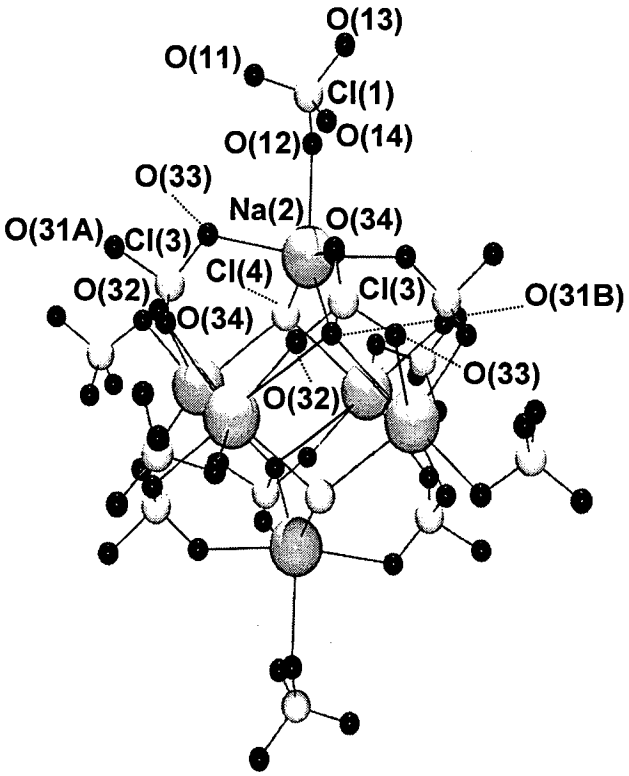


Figure S1. Crystal structure of cluster **10**

Table 1. Crystal data and structure refinement for $[(Na(ClO_4)_6)\subset \mathbf{L1}_2H_{13}]\ Na_6Cl_2(ClO_4)_{12}$

Identification code	$[(Na(ClO_4)_6)\subset \mathbf{L1}_2H_{13}][Na_6Cl_2(ClO_4)_{12}]$
Empirical formula	C60 H120 Cl20 N26 Na7 O72
Formula weight	3227.76
Temperature	100(2) K
Wavelength	1.54178 A
Crystal system, space group	Trigonal, R -3
Unit cell dimensions	a = 21.669(2) A alpha = 90 deg. b = 21.670(2) A beta = 90 deg. c = 23.534(2) A gamma = 120 deg.
Volume	9570.3(15) A^3
Z, Calculated density	3, 1.680 Mg/m^3
Absorption coefficient	5.161 mm^-1
F(000)	4965
Crystal size	1.00 x 0.3 x 0.3 mm
Theta range for data collection	4.18 to 57.95 deg.
Limiting indices	-23<=h<=22, -23<=k<=20, -24<=l<=23
Reflections collected / unique	9707 / 2603 [R(int) = 0.0593]
Completeness to theta = 57.95	94.6 %
Absorption correction	Analytical
Max. and min. transmission	0.213 and 0.112
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2603 / 0 / 289
Goodness-of-fit on F^2	0.972
Final R indices [I>2sigma(I)]	R1 = 0.0834, wR2 = 0.2233
R indices (all data)	R1 = 0.0938, wR2 = 0.2342
Extinction coefficient	0.000019(18)
Largest diff. peak and hole	1.235 and -0.982 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for $[(Na(ClO_4)_6)\subset \mathbf{L1}_2H_{13}]Na_6Cl_2(ClO_4)_{12}$. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Cl(1)	5815(1)	703(1)	1530(1)	32(1)
O(11)	6461(3)	749(4)	1380(3)	62(2)
O(12)	5876(4)	1377(4)	1494(6)	143(5)
O(13)	5217(4)	198(5)	1195(3)	77(2)
O(14)	5664(4)	496(6)	2106(4)	108(3)
Cl(2)	3849(1)	-1766(1)	886(1)	29(1)
O(21)	3518(3)	-1339(3)	946(2)	35(1)
O(22)	4305(3)	-1533(3)	395(2)	43(2)
O(23)	3289(3)	-2501(3)	795(3)	43(2)
O(24)	4242(3)	-1730(4)	1382(3)	50(2)
O(31B)	7444(10)	2793(10)	3106(8)	51(5)
Cl(3)	8034(2)	2582(1)	2782(1)	63(1)
O(31A)	8626(5)	2452(5)	2559(4)	51(2)
O(32)	8277(5)	3244(5)	2522(4)	92(3)
O(33)	7437(5)	2034(6)	2523(6)	145(5)
O(34)	8154(9)	2565(9)	3343(5)	171(6)
Cl(4)	6667	3333	2122(2)	73(1)
Na(1)	3333	-3333	1667	44(2)
Na(2)	6220(2)	2003(2)	2550(2)	64(1)
N(1)	3333	-3333	-852(4)	16(2)
C(1)	3995(3)	-2680(3)	-641(3)	17(2)
C(2)	4633(3)	-2509(4)	-1014(3)	20(2)
N(2)	5294(3)	-2055(3)	-696(2)	17(1)
C(3)	5893(4)	-1637(4)	-1086(3)	23(2)
C(4)	5869(4)	-995(4)	-1312(3)	28(2)
N(3)	5836(3)	-569(3)	-823(3)	27(2)
C(5)	6544(4)	-74(4)	-557(3)	32(2)
C(6)	6405(5)	158(4)	8(4)	40(2)
N(4)	5963(3)	-464(3)	366(2)	25(2)
C(7)	6382(4)	-654(4)	744(3)	32(2)
C(8)	5907(4)	-1361(4)	1019(3)	29(2)
N(5)	5470(3)	-1904(3)	567(2)	21(1)
C(9)	5902(4)	-2092(4)	184(3)	23(2)
C(10)	5466(4)	-2494(3)	-324(3)	22(2)

Table 3. Bond lengths [A] and angles [deg] for $[(Na(ClO_4)_6)\subset \mathbf{L1}_2H_{13}]Na_6Cl_2(ClO_4)_{12}$

Cl(1)-O(11)	1.398(6)
Cl(1)-O(12)	1.402(8)
Cl(1)-O(14)	1.414(9)
Cl(1)-O(13)	1.442(7)
O(12)-Na(2)	2.749(15)
Cl(2)-O(24)	1.424(6)
Cl(2)-O(21)	1.432(5)
Cl(2)-O(22)	1.437(6)
Cl(2)-O(23)	1.457(6)
O(23)-Na(1)	2.766(6)
O(31B)-Cl(3)	1.737(18)
O(31B)-Na(2)#1	2.388(18)
O(31B)-Na(2)	2.67(2)
O(31B)-Na(2)#2	2.996(18)
Cl(3)-O(34)	1.351(11)
Cl(3)-O(33)	1.386(11)
Cl(3)-O(32)	1.398(8)
Cl(3)-O(31A)	1.536(9)
O(32)-Na(2)#2	2.470(9)
O(33)-Na(2)	2.604(13)
O(34)-Na(2)#1	2.533(13)
Cl(4)-Na(2)#2	2.734(5)
Cl(4)-Na(2)#3	2.734(5)
Cl(4)-Na(2)	2.734(5)
Na(1)-O(23)#4	2.766(6)
Na(1)-O(23)#5	2.766(6)
Na(1)-O(23)#6	2.766(6)
Na(1)-O(23)#7	2.766(6)
Na(1)-O(23)#8	2.766(6)
Na(2)-O(31B)#9	2.388(18)
Na(2)-O(32)#3	2.470(9)
Na(2)-O(34)#9	2.533(13)
Na(2)-O(31B)#3	2.996(18)
N(1)-C(1)#5	1.508(7)
N(1)-C(1)	1.508(7)
N(1)-C(1)#4	1.508(7)
C(1)-C(2)	1.518(9)
C(2)-N(2)	1.474(9)
N(2)-C(3)	1.474(9)
N(2)-C(10)	1.471(9)
C(3)-C(4)	1.513(10)
C(4)-N(3)	1.501(9)
N(3)-C(5)	1.501(10)
C(5)-C(6)	1.505(11)
C(6)-N(4)	1.468(10)
N(4)-C(7)	1.469(9)
C(7)-C(8)	1.499(11)
C(8)-N(5)	1.516(9)
N(5)-C(9)	1.496(9)
C(9)-C(10)	1.502(10)

O(11)-Cl(1)-O(12)	109.9(5)
O(11)-Cl(1)-O(14)	109.2(5)
O(12)-Cl(1)-O(14)	106.0(8)
O(11)-Cl(1)-O(13)	113.8(4)
O(12)-Cl(1)-O(13)	109.7(6)
O(14)-Cl(1)-O(13)	107.9(5)

Cl(1)-O(12)-Na(2)	108.7(7)
O(24)-Cl(2)-O(21)	111.2(4)
O(24)-Cl(2)-O(22)	110.6(4)
O(21)-Cl(2)-O(22)	109.4(3)
O(24)-Cl(2)-O(23)	109.2(4)
O(21)-Cl(2)-O(23)	107.9(3)
O(22)-Cl(2)-O(23)	108.5(3)
Cl(2)-O(23)-Na(1)	108.8(3)
Cl(3)-O(31B)-Na(2)#1	114.0(9)
Cl(3)-O(31B)-Na(2)	104.0(9)
Na(2)#1-O(31B)-Na(2)	124.4(7)
Cl(3)-O(31B)-Na(2)#2	96.4(7)
Na(2)#1-O(31B)-Na(2)#2	112.1(7)
Na(2)-O(31B)-Na(2)#2	101.8(6)
O(34)-Cl(3)-O(33)	121.1(10)
O(34)-Cl(3)-O(32)	118.4(8)
O(33)-Cl(3)-O(32)	113.4(8)
O(34)-Cl(3)-O(31A)	98.1(7)
O(33)-Cl(3)-O(31A)	101.6(6)
O(32)-Cl(3)-O(31A)	96.8(5)
O(34)-Cl(3)-O(31B)	75.7(8)
O(33)-Cl(3)-O(31B)	85.6(8)
O(32)-Cl(3)-O(31B)	82.5(7)
O(31A)-Cl(3)-O(31B)	172.3(8)
Cl(3)-O(32)-Na(2)#2	137.6(6)
Cl(3)-O(33)-Na(2)	120.8(6)
Cl(3)-O(34)-Na(2)#1	124.3(8)
Na(2)#2-Cl(4)-Na(2)#3	107.22(14)
Na(2)#2-Cl(4)-Na(2)	107.23(14)
Na(2)#3-Cl(4)-Na(2)	107.23(14)
O(23)#4-Na(1)-O(23)#5	71.00(19)
O(23)#4-Na(1)-O(23)#6	109.00(19)
O(23)#5-Na(1)-O(23)#6	108.99(19)
O(23)#4-Na(1)-O(23)	71.01(19)
O(23)#5-Na(1)-O(23)	71.01(19)
O(23)#6-Na(1)-O(23)	180.0
O(23)#4-Na(1)-O(23)#7	109.00(19)
O(23)#5-Na(1)-O(23)#7	180.0
O(23)#6-Na(1)-O(23)#7	71.01(19)
O(23)-Na(1)-O(23)#7	108.99(19)
O(23)#4-Na(1)-O(23)#8	180.0
O(23)#5-Na(1)-O(23)#8	109.00(19)
O(23)#6-Na(1)-O(23)#8	71.00(19)
O(23)-Na(1)-O(23)#8	108.99(19)
O(23)#7-Na(1)-O(23)#8	71.00(19)
O(31B)#9-Na(2)-O(32)#3	86.6(5)
O(31B)#9-Na(2)-O(34)#9	45.8(5)
O(32)#3-Na(2)-O(34)#9	89.5(5)
O(31B)#9-Na(2)-O(33)	96.6(6)
O(32)#3-Na(2)-O(33)	168.6(4)
O(34)#9-Na(2)-O(33)	85.0(5)
O(31B)#9-Na(2)-O(31B)	64.4(5)
O(32)#3-Na(2)-O(31B)	141.9(5)
O(34)#9-Na(2)-O(31B)	87.0(5)
O(33)-Na(2)-O(31B)	47.8(5)
O(31B)#9-Na(2)-Cl(4)	110.0(5)
O(32)#3-Na(2)-Cl(4)	90.8(3)
O(34)#9-Na(2)-Cl(4)	155.7(3)
O(33)-Na(2)-Cl(4)	98.3(3)
O(31B)-Na(2)-Cl(4)	77.9(4)
O(31B)#9-Na(2)-O(12)	154.4(5)
O(32)#3-Na(2)-O(12)	82.0(3)

O(34)#9-Na(2)-O(12)	111.1(4)
O(33)-Na(2)-O(12)	90.7(3)
O(31B)-Na(2)-O(12)	134.2(4)
Cl(4)-Na(2)-O(12)	93.0(2)
O(31B)#9-Na(2)-O(31B)#3	59.1(5)
O(32)#3-Na(2)-O(31B)#3	43.5(4)
O(34)#9-Na(2)-O(31B)#3	91.3(6)
O(33)-Na(2)-O(31B)#3	146.4(5)
O(31B)-Na(2)-O(31B)#3	98.7(7)
Cl(4)-Na(2)-O(31B)#3	72.6(4)
O(12)-Na(2)-O(31B)#3	121.5(4)
C(1)#5-N(1)-C(1)	109.8(4)
C(1)#5-N(1)-C(1)#4	109.8(4)
C(1)-N(1)-C(1)#4	109.8(4)
N(1)-C(1)-C(2)	111.2(6)
N(2)-C(2)-C(1)	109.4(5)
C(2)-N(2)-C(3)	110.9(5)
C(2)-N(2)-C(10)	110.3(5)
C(3)-N(2)-C(10)	109.8(5)
N(2)-C(3)-C(4)	111.1(6)
N(3)-C(4)-C(3)	109.2(6)
C(4)-N(3)-C(5)	114.4(6)
C(6)-C(5)-N(3)	107.6(6)
N(4)-C(6)-C(5)	110.3(6)
C(7)-N(4)-C(6)	113.3(6)
N(4)-C(7)-C(8)	110.0(6)
C(7)-C(8)-N(5)	109.4(6)
C(9)-N(5)-C(8)	113.7(5)
N(5)-C(9)-C(10)	109.9(5)
N(2)-C(10)-C(9)	112.8(5)

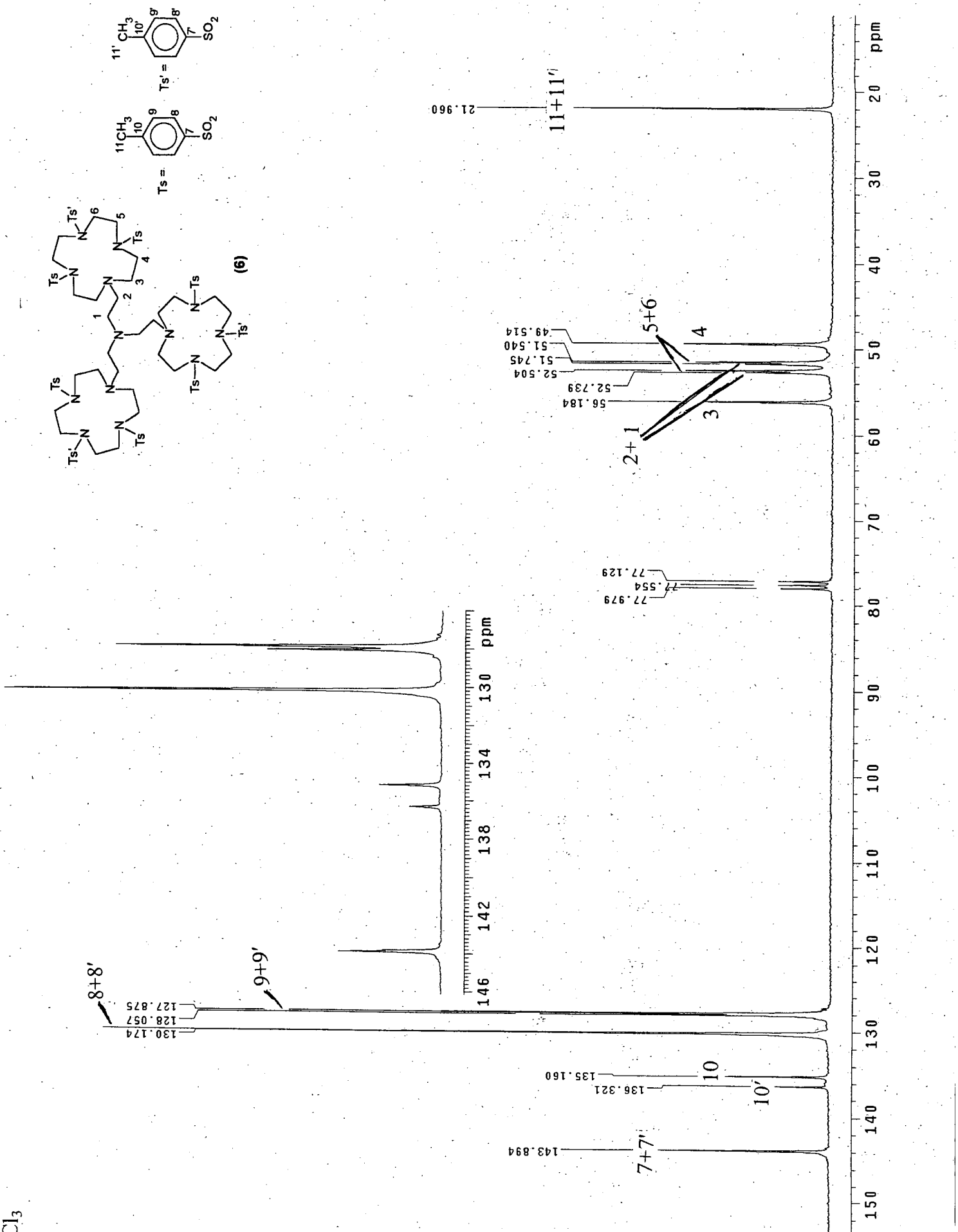
Symmetry transformations used to generate equivalent atoms:
#1 x-y+1/3,x-1/3,-z+2/3 #2 -y+1,x-y,z
#3 -x+y+1,-x+1,z #4 -x+y+1,-x,z #5 -y,x-y-1,z
#6 -x+2/3,-y-2/3,-z+1/3 #7 y+2/3,-x+y+1/3,-z+1/3
#8 x-y-1/3,x-2/3,-z+1/3 #9 y+1/3,-x+y+2/3,-z+2/3

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for $[(Na(ClO_4)_6)\subset \mathbf{L1}_2H_{13}]Na_6Cl_2(ClO_4)_{12}$.
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
Cl(1)	33(1)	28(1)	40(1)	7(1)	11(1)	19(1)
O(11)	50(4)	82(5)	69(5)	-9(4)	0(3)	44(4)
O(12)	53(5)	41(5)	338(18)	42(7)	27(7)	27(4)
O(13)	60(5)	115(6)	80(5)	-39(5)	-28(4)	63(5)
O(14)	62(5)	145(9)	67(6)	19(5)	4(4)	13(5)
Cl(2)	20(1)	39(1)	33(1)	-4(1)	-4(1)	19(1)
O(21)	39(3)	43(3)	38(3)	2(3)	0(2)	32(3)
O(22)	32(3)	49(4)	47(4)	-5(3)	10(3)	21(3)
O(23)	28(3)	34(3)	63(4)	0(3)	3(3)	12(3)
O(24)	38(3)	75(4)	48(4)	-2(3)	-18(3)	36(3)
O(31B)	52(11)	66(13)	40(11)	18(9)	13(9)	34(10)
Cl(3)	92(2)	53(2)	40(2)	4(1)	-9(1)	33(2)
O(31A)	64(6)	42(5)	63(6)	9(5)	13(5)	7(5)
O(32)	125(7)	83(6)	85(6)	18(5)	4(5)	66(6)
O(33)	84(7)	106(8)	188(12)	-48(8)	50(7)	5(6)
O(34)	295(18)	247(16)	64(7)	43(8)	34(9)	204(16)
Cl(4)	77(2)	77(2)	67(3)	0	0	38(1)
Na(1)	44(3)	44(3)	44(5)	0	0	22(2)
Na(2)	50(2)	88(3)	54(2)	7(2)	1(2)	35(2)
N(1)	13(3)	13(3)	21(5)	0	0	6(2)
C(1)	17(4)	14(3)	23(4)	-2(3)	0(3)	10(3)
C(2)	16(4)	16(4)	25(4)	-4(3)	0(3)	7(3)
N(2)	13(3)	13(3)	23(3)	-1(2)	1(2)	6(2)
C(3)	19(4)	19(4)	28(4)	-2(3)	6(3)	6(3)
C(4)	26(4)	28(4)	21(4)	3(3)	3(3)	6(3)
N(3)	33(4)	25(3)	28(4)	1(3)	2(3)	19(3)
C(5)	29(4)	8(4)	42(5)	3(3)	10(4)	-3(3)
C(6)	45(5)	15(4)	46(5)	-8(4)	0(4)	5(4)
N(4)	26(3)	12(3)	28(4)	2(2)	-2(3)	2(3)
C(7)	21(4)	26(4)	43(5)	-10(4)	-11(4)	7(3)
C(8)	30(4)	38(5)	24(4)	-2(3)	-4(3)	21(4)
N(5)	16(3)	19(3)	28(3)	-2(3)	-2(2)	9(3)
C(9)	19(4)	18(4)	39(5)	5(3)	2(3)	14(3)
C(10)	18(4)	13(4)	38(4)	-2(3)	1(3)	9(3)

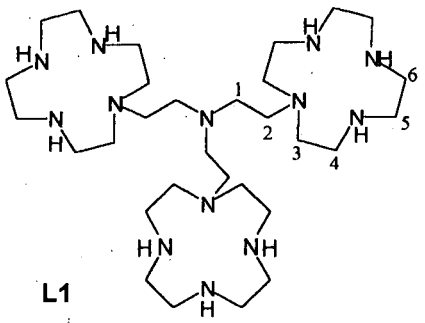
Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for $[(Na(ClO_4)_6)\subset \mathbf{L1}_2H_{13}]Na_6Cl_2(ClO_4)_{12}$

	x	y	z	U(eq)
H(1A)	4093	-2759	-254	21
H(1B)	3915	-2278	-640	21
H(2A)	4617	-2947	-1128	24
H(2B)	4620	-2263	-1355	24
H(3A)	5872	-1936	-1401	28
H(3B)	6339	-1479	-888	28
H(4A)	6291	-703	-1539	34
H(4B)	5454	-1150	-1553	34
H(5A)	6815	-316	-505	38
H(5B)	6816	336	-800	38
H(6A)	6854	465	197	48
H(6B)	6163	428	-51	48
H(7A)	6612	-291	1034	38
H(7B)	6750	-678	527	38
H(8A)	5592	-1316	1288	35
H(8B)	6193	-1517	1224	35
H(9A)	6053	-2382	389	28
H(9B)	6325	-1660	60	28
H(10A)	5727	-2670	-541	27
H(10B)	5026	-2903	-194	27

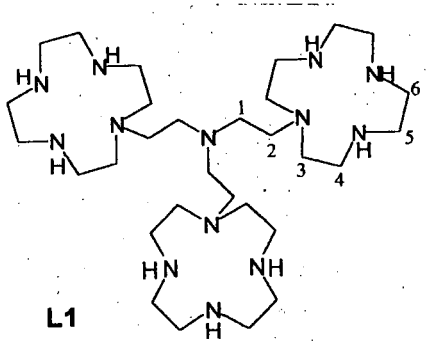


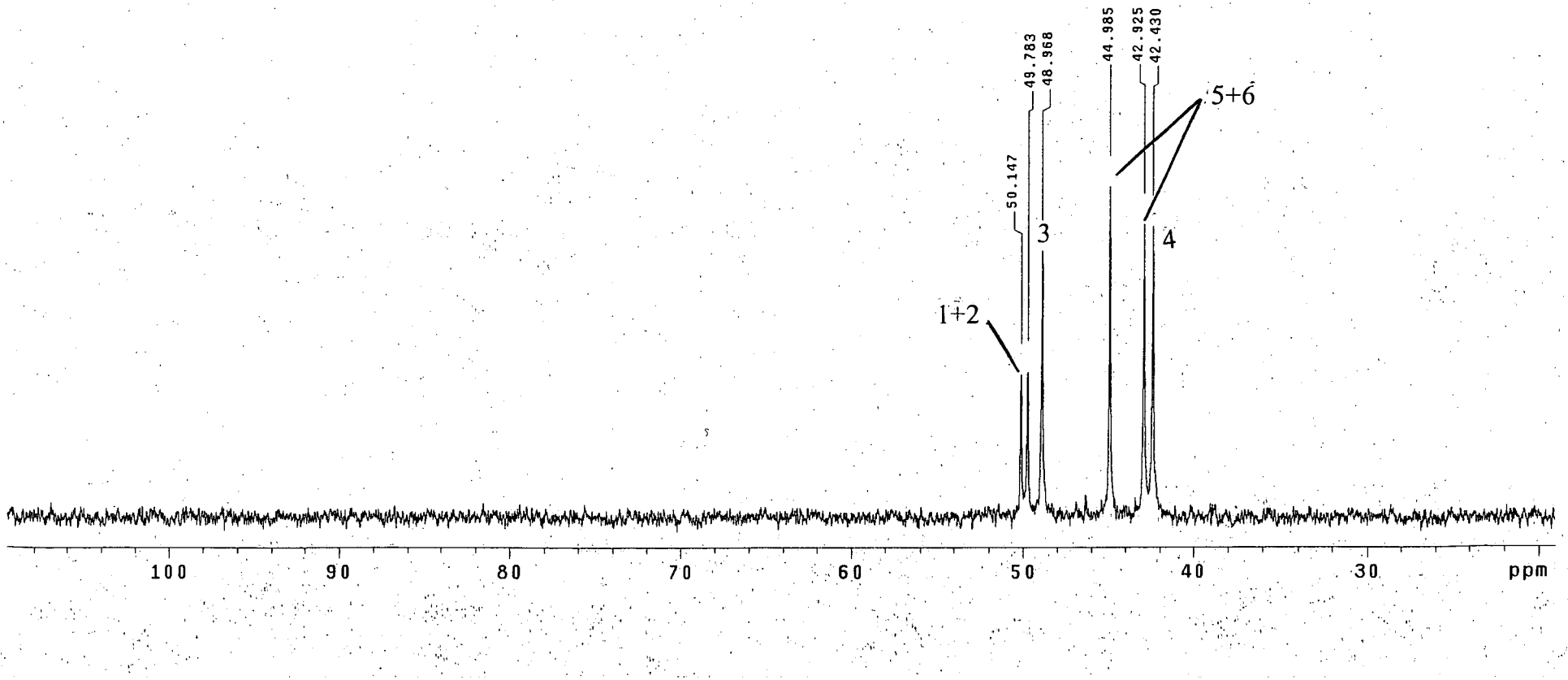
^{13}C NMR spectrum of compound (6)
$CDCl_3$

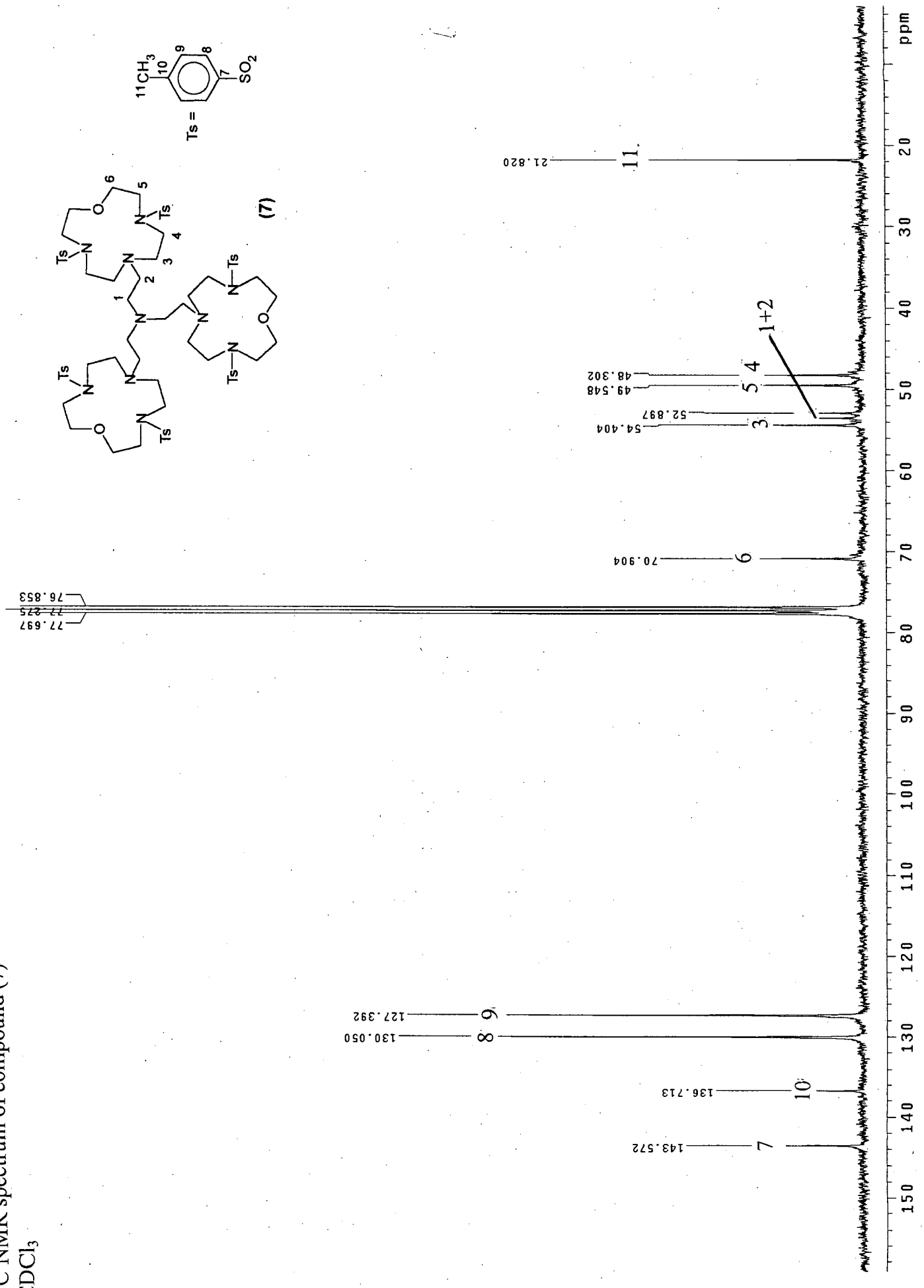
H NMR spectrum of compound L1
$_2$O pH = 8.3



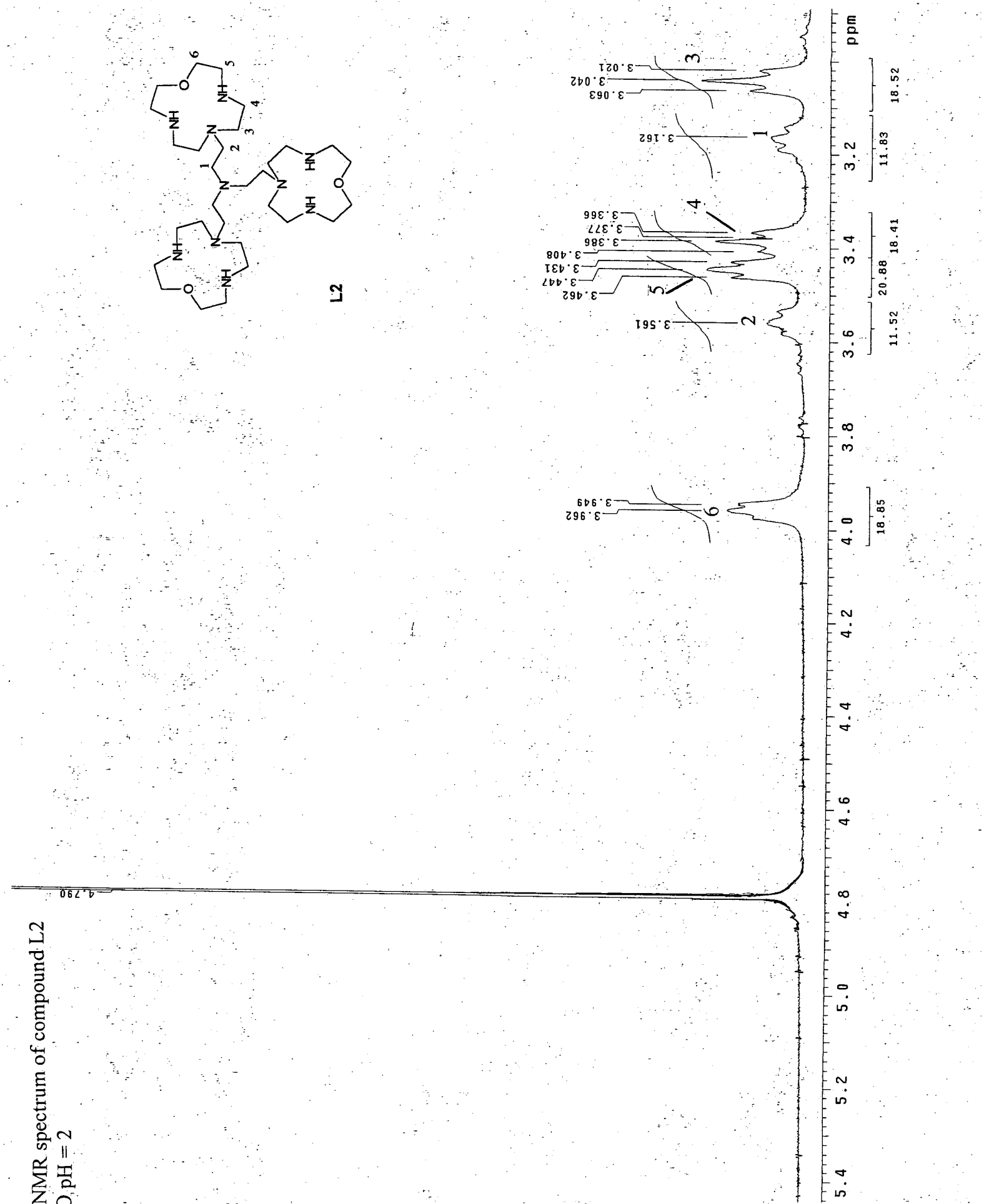
^{13}C NMR spectrum of compound L1
D_2O pH = 8.3





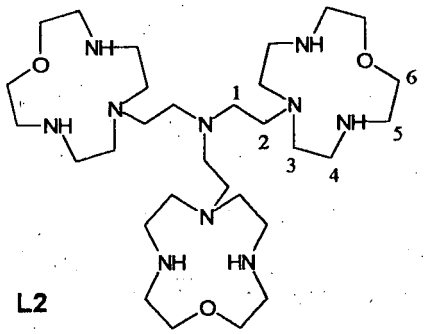


^{13}C NMR spectrum of compound (7)
$CDCl_3$

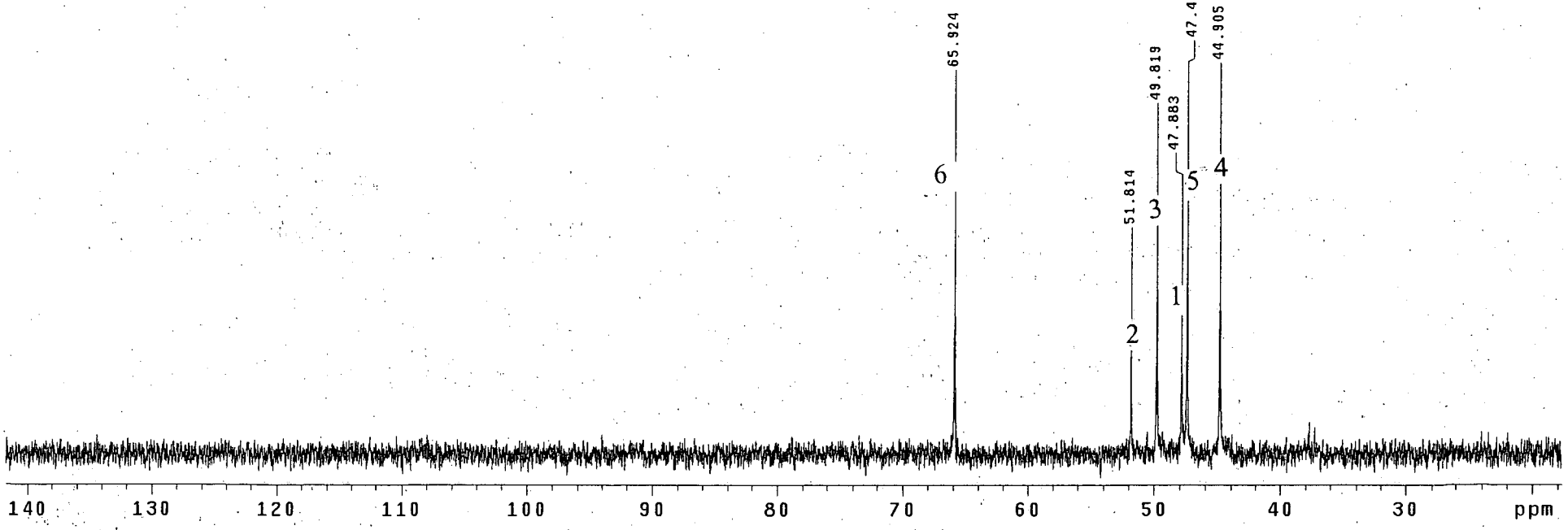


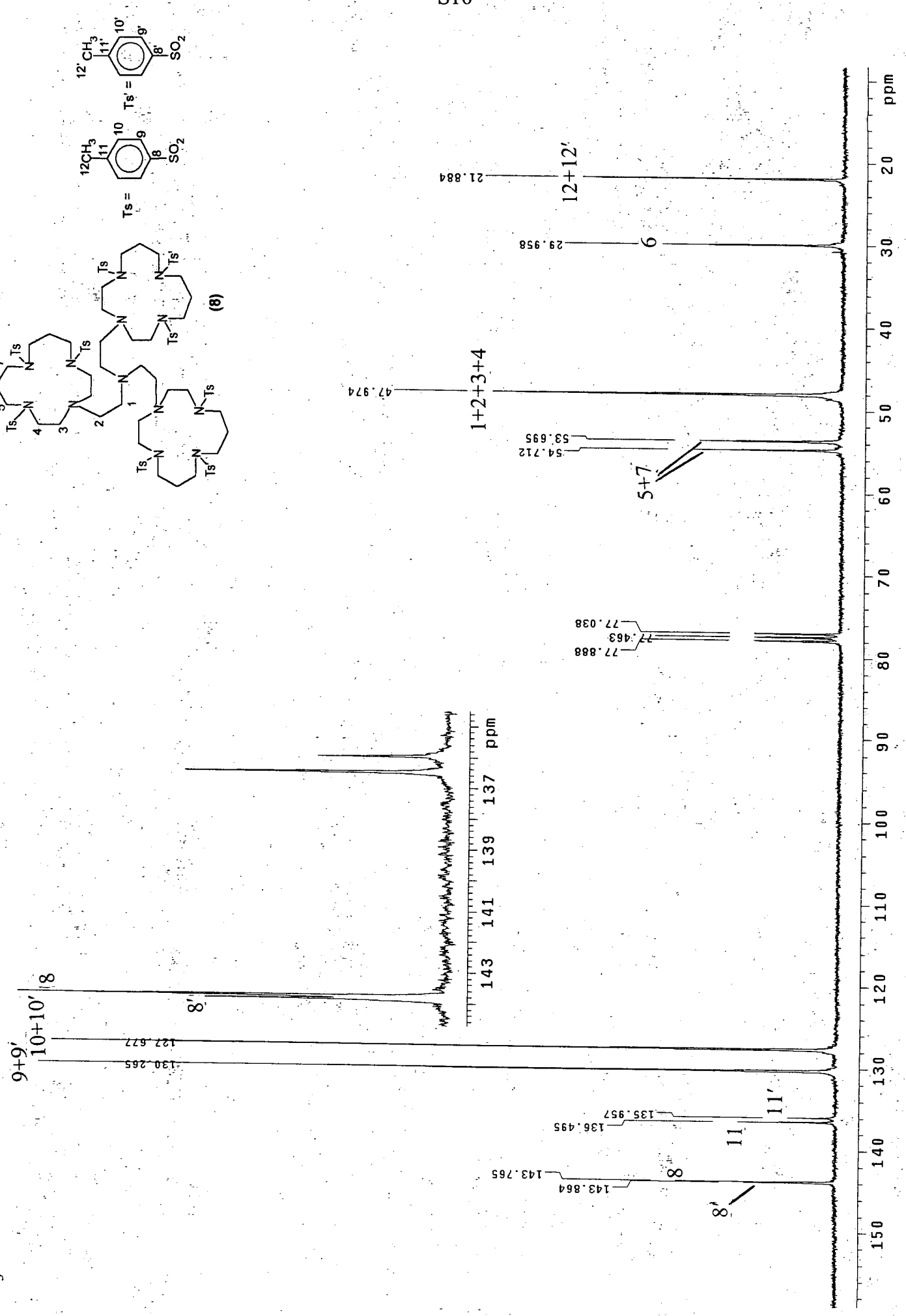
H NMR spectrum of compound L2
$_2$O, pH = 2

^{13}C NMR spectrum of compound L2
D_2O pH = 2

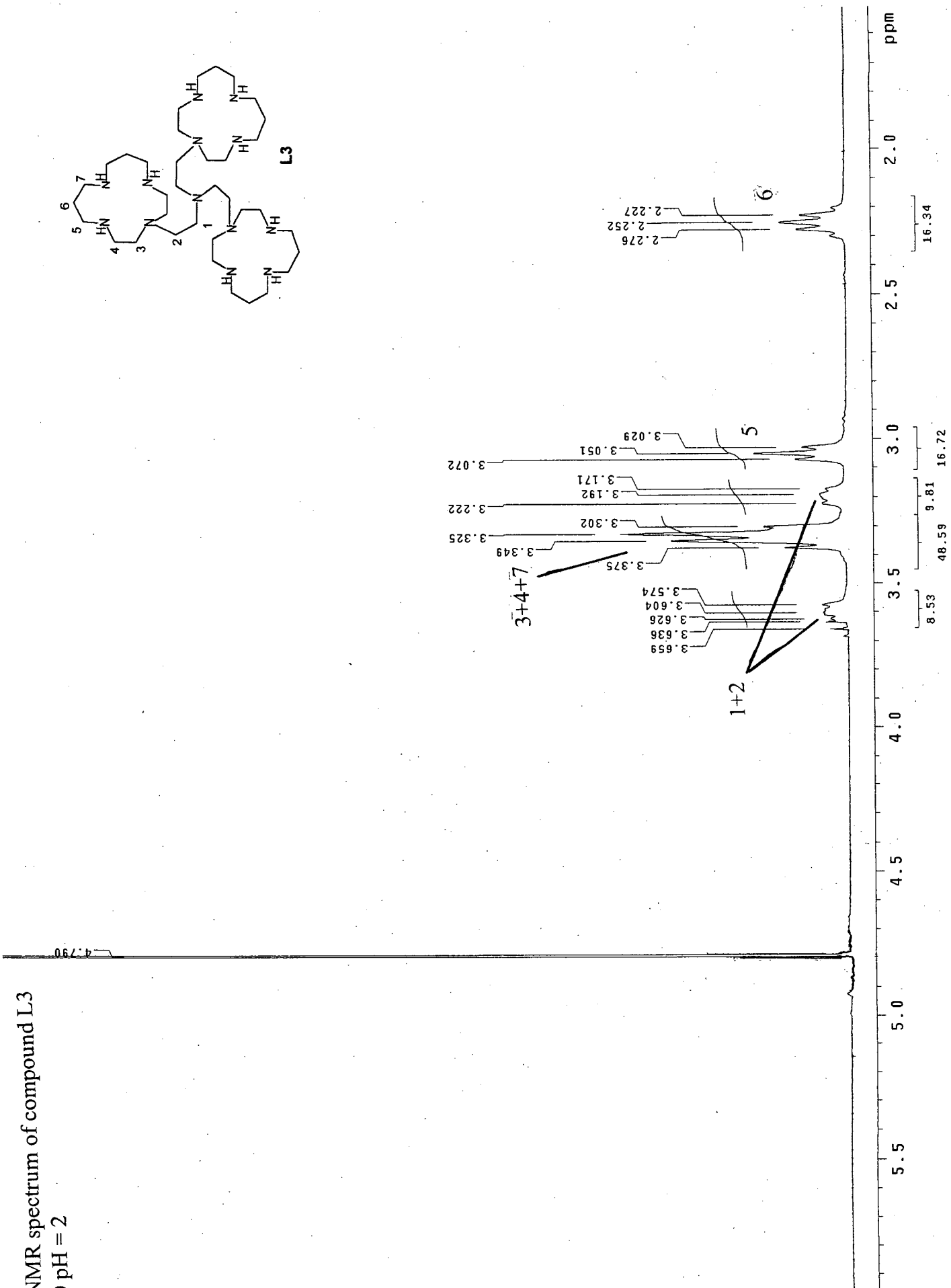


L2





^{13}C NMR spectrum of compound (8)
$CDCl_3$



H NMR spectrum of compound L3
D_2O pH = 2

^{13}C NMR spectrum of compound L3
D_2O pH = 2