APPENDIX D

Crystallographic Data for TBA·Ln(DO2A)(DPA) Structures

CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Date 26 July 2007

Crystal Structure Analysis of:

MLC07

(shown below)

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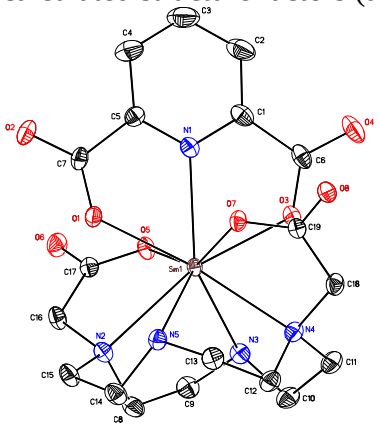


MLC07

Note: The crystallographic data have been deposited in the Cambridge Database (CCDC) and has been placed on hold pending further instructions from me. The deposition number is 655647. Ideally the CCDC would like the publication to contain a footnote of the type: "Crystallographic data have been deposited at the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK and copies can be obtained on request, free of charge, by quoting the publication citation and the deposition number 655647."

Table 1. Crystal data and structure refinement for MLC07 (CCDC 655647).

Empirical formula	$[C_{19}H_{25}N_5O_8Sm]^-$ $[C_{16}H_{36}N]^+$ $2(H_2O)$, $0.73(C_3H_6O)$, $0.27(C_2H_6O \cdot O)$
Formula weight	939.36
Crystallization Solvent	Acetone/ethanol/isopropanol
Crystal Habit	Plate
Crystal size	0.33 x 0.24 x 0.11 mm^3
Crystal color	Yellow

Data Collection

Type of diffractometer	Bruker SMART 1000
Wavelength	0.71073 Å MoKα
Data Collection Temperature	100(2) K
θ range for 33380 reflections used in lattice determination	2.17 to 39.91°
Unit cell dimensions	a = 13.0658(4) Å b = 13.4504(4) Å β= 90.3240(10)° c = 26.1778(7) Å
Volume	4600.4(2) $Å^3$
Z	4
Crystal system	Monoclinic
Space group	$P2_1/c$
Density (calculated)	1.356 Mg/m^3
F(000)	1966
θ range for data collection	1.70 to 40.79°
Completeness to θ = 40.79°	94.6 %
Index ranges	$-23 \le h \le 23$, $-22 \le k \le 24$, $-47 \le l \le 44$
Data collection scan type	ω scans at 7 ϕ settings
Reflections collected	150100
Independent reflections	28270 [R_{int}= 0.1100]
Absorption coefficient	1.336 mm^{-1}
Absorption correction	None
Max. and min. transmission	0.8670 and 0.6669

Table 1 (cont.)

Structure solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 1990)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	SHELXL-97 (Sheldrick, 1997)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	28270 / 6 / 560
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	1.124
Final R indices [$I>2\sigma(I)$, 14923 reflections]	R1 = 0.0422, *w*R2 = 0.0734
R indices (all data)	R1 = 0.0997, *w*R2 = 0.0821
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.009
Average shift/error	0.000
Largest diff. peak and hole	3.079 and -2.442 e.Å^{-3}

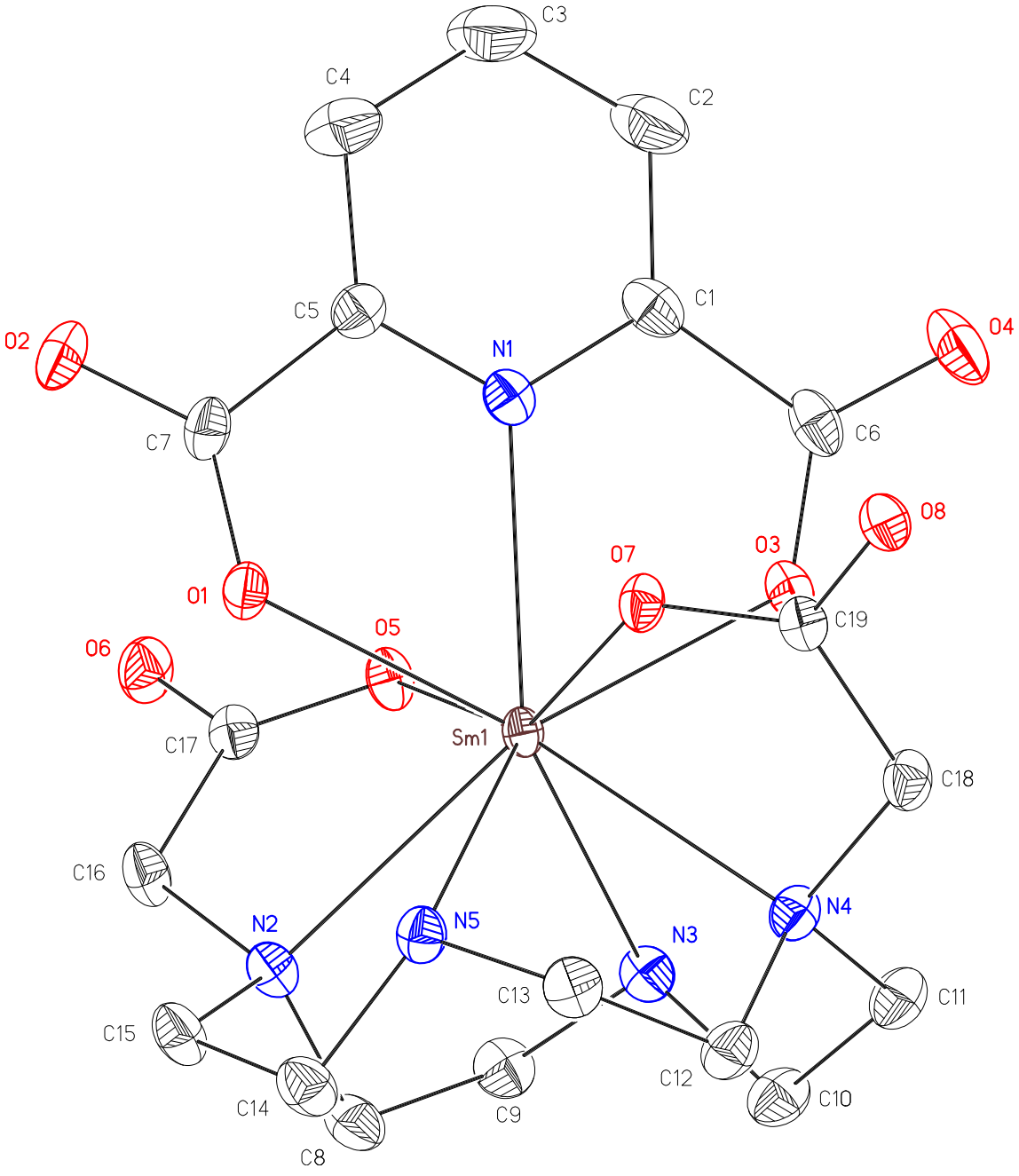
Special Refinement Details

The solvent region is disordered, containing acetone (73%) and ethanol/water (27%). These were refined without restraint. Additionally, the solvent region contains two ordered water molecules which were refined with hydrogen geometry restrained.

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

D5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O6
Sm1
C17
C18
C16
N2
N5
N3
N4
C13
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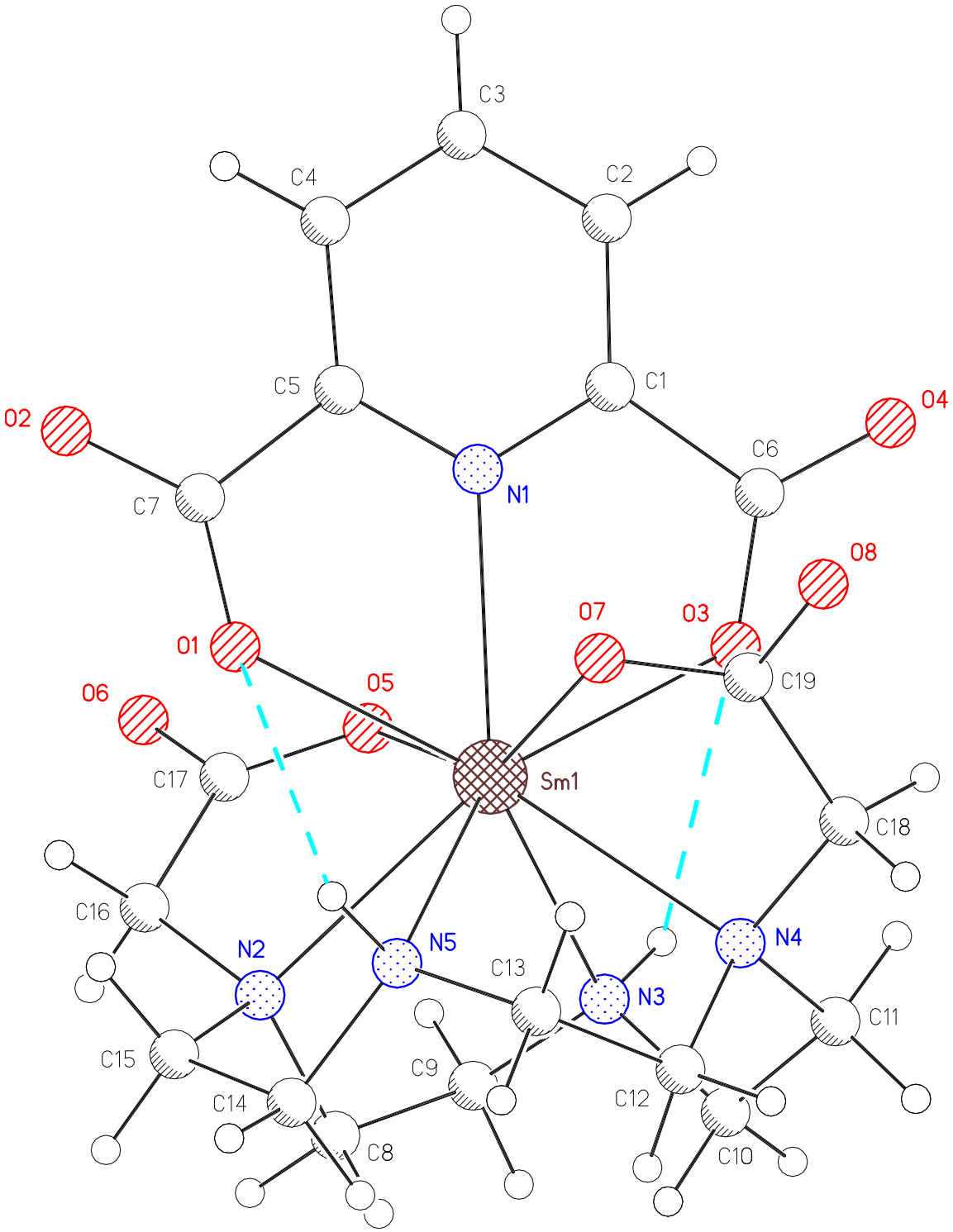
C15
C12
C14
C10
C9
C8

D6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O6
C17
Sm1
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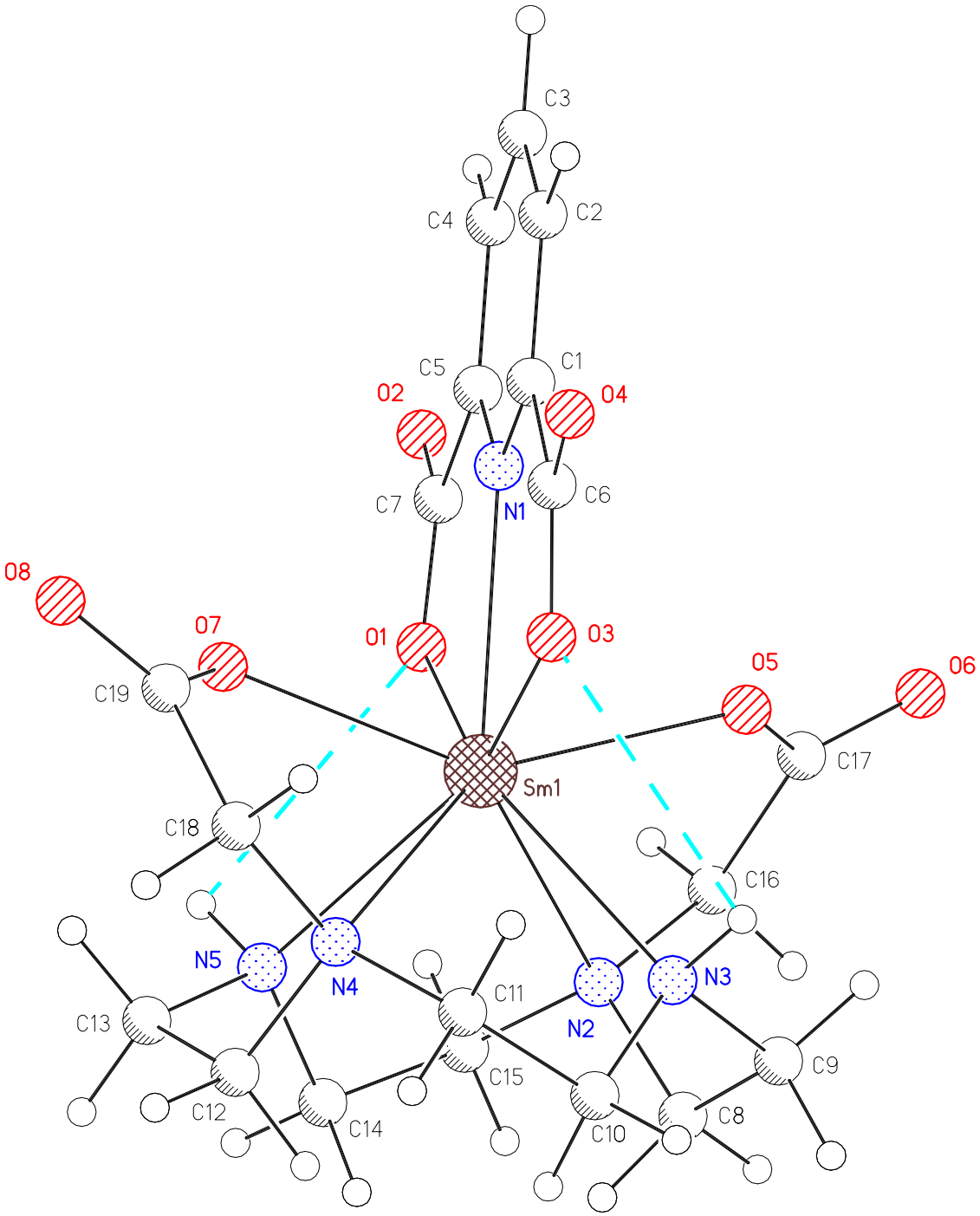
C11
C15
C9
C12
C14
C8
C10

D7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C19
Sm1
C17
C18
C16
N5
N4
N3
C11
N2
C13
C9
C15
C12
C14
C10
C8

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for MLC07 (CCDC 655647). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}	Occ
Sm(1)	7494(1)	4931(1)	5001(1)	12(1)	1
O(1)	8313(1)	5490(1)	5785(1)	17(1)	1
O(2)	8658(2)	6668(1)	6366(1)	33(1)	1
O(3)	6717(1)	5941(1)	4337(1)	18(1)	1
O(4)	6445(1)	7418(1)	3952(1)	30(1)	1
O(5)	6038(1)	5292(1)	5515(1)	16(1)	1
O(6)	5302(1)	5278(1)	6280(1)	22(1)	1
O(7)	8982(1)	5576(1)	4585(1)	16(1)	1
O(8)	9678(1)	6092(1)	3855(1)	17(1)	1
N(1)	7516(1)	6789(1)	5149(1)	15(1)	1
N(2)	7032(1)	3512(1)	5682(1)	17(1)	1
N(3)	6029(1)	3789(1)	4676(1)	17(1)	1
N(4)	7949(1)	4027(1)	4121(1)	16(1)	1
N(5)	8939(1)	3607(1)	5116(1)	16(1)	1
C(1)	7140(2)	7403(2)	4792(1)	20(1)	1
C(2)	7132(2)	8426(2)	4863(1)	31(1)	1
C(3)	7519(2)	8808(2)	5318(1)	38(1)	1
C(4)	7901(2)	8169(2)	5686(1)	28(1)	1
C(5)	7893(2)	7152(2)	5583(1)	17(1)	1
C(6)	6733(2)	6888(2)	4315(1)	19(1)	1
C(7)	8319(2)	6373(2)	5948(1)	18(1)	1
C(8)	6332(2)	2770(2)	5449(1)	22(1)	1
C(9)	5538(2)	3231(2)	5096(1)	20(1)	1
C(10)	6284(2)	3149(2)	4236(1)	21(1)	1
C(11)	6975(2)	3701(2)	3870(1)	20(1)	1
C(12)	8624(2)	3161(2)	4212(1)	20(1)	1
C(13)	9426(2)	3352(2)	4624(1)	19(1)	1
C(14)	8670(2)	2724(2)	5418(1)	21(1)	1
C(15)	7985(2)	3011(2)	5856(1)	21(1)	1
C(16)	6535(2)	4022(2)	6114(1)	19(1)	1
C(17)	5899(1)	4923(2)	5959(1)	15(1)	1
C(18)	8457(2)	4782(2)	3800(1)	18(1)	1
C(19)	9099(1)	5537(1)	4103(1)	14(1)	1
N(6)	8625(1)	7115(1)	7812(1)	18(1)	1
C(21)	9621(2)	6949(2)	7523(1)	20(1)	1
C(22)	10552(2)	7499(2)	7733(1)	24(1)	1
C(23)	11416(2)	7497(2)	7342(1)	25(1)	1
C(24)	12372(2)	8025(2)	7542(1)	34(1)	1
C(25)	8744(2)	6830(2)	8371(1)	20(1)	1
C(26)	9016(2)	5745(2)	8471(1)	31(1)	1
C(27)	9270(3)	5576(2)	9024(1)	42(1)	1
C(28)	9436(3)	4504(2)	9174(2)	57(1)	1
C(29)	7815(2)	6478(2)	7547(1)	22(1)	1
C(30)	6729(2)	6619(2)	7742(1)	26(1)	1
C(31)	6007(2)	5924(3)	7458(1)	48(1)	1
C(32)	4887(2)	6117(3)	7596(1)	55(1)	1

C(33)	8325(2)	8203(2)	7802(1)	20(1)	1
C(34)	8166(2)	8649(2)	7271(1)	26(1)	1
C(35)	7925(2)	9747(2)	7314(1)	27(1)	1
C(36)	7715(2)	10218(2)	6793(1)	35(1)	1
O(21)	4163(1)	3901(1)	6866(1)	37(1)	1
O(22)	746(1)	5096(1)	3092(1)	26(1)	1
O(51)	3632(3)	9306(2)	5430(2)	69(1)	0.731(3)
C(51)	5010(4)	10204(4)	5845(2)	67(2)	0.731(3)
C(52)	4046(5)	9525(3)	5821(2)	54(1)	0.731(3)
C(53)	3596(5)	9324(4)	6253(3)	83(2)	0.731(3)
O(61)	5133(6)	8689(5)	7101(3)	40(2)	0.269(3)
C(62)	4947(11)	8807(10)	6546(6)	62(4)	0.269(3)
C(64)	4410(11)	9466(11)	6164(9)	91(8)	0.269(3)
O(71)	6114(5)	7249(5)	6485(2)	29(2)	0.269(3)

Table 3. Bond lengths [Å] and angles [°] for MLC07 (CCDC 655647).

Sm(1)-O(5)	2.3858(14)	C(27)-C(28)	1.510(4)
Sm(1)-O(7)	2.3964(14)	C(29)-C(30)	1.522(3)
Sm(1)-O(3)	2.4246(14)	C(30)-C(31)	1.519(3)
Sm(1)-O(1)	2.4283(14)	C(31)-C(32)	1.532(4)
Sm(1)-N(1)	2.5295(17)	C(33)-C(34)	1.527(3)
Sm(1)-N(3)	2.5940(17)	C(34)-C(35)	1.514(3)
Sm(1)-N(5)	2.6115(16)	C(35)-C(36)	1.528(3)
Sm(1)-N(4)	2.6740(16)	O(51)-C(52)	1.191(6)
Sm(1)-N(2)	2.6824(16)	C(51)-C(52)	1.556(8)
O(1)-C(7)	1.263(2)	C(52)-C(53)	1.306(8)
O(2)-C(7)	1.243(2)	O(61)-C(62)	1.481(17)
O(3)-C(6)	1.274(3)	C(62)-C(64)	1.51(2)
O(4)-C(6)	1.245(2)		
O(5)-C(17)	1.278(2)	O(5)-Sm(1)-O(7)	146.52(6)
O(6)-C(17)	1.245(2)	O(5)-Sm(1)-O(3)	87.60(5)
O(7)-C(19)	1.272(2)	O(7)-Sm(1)-O(3)	78.96(5)
O(8)-C(19)	1.247(2)	O(5)-Sm(1)-O(1)	79.02(5)
N(1)-C(5)	1.330(3)	O(7)-Sm(1)-O(1)	85.28(5)
N(1)-C(1)	1.339(3)	O(3)-Sm(1)-O(1)	127.82(5)
N(2)-C(16)	1.477(3)	O(5)-Sm(1)-N(1)	73.79(5)
N(2)-C(8)	1.482(3)	O(7)-Sm(1)-N(1)	72.75(5)
N(2)-C(15)	1.484(3)	O(3)-Sm(1)-N(1)	63.90(5)
N(3)-C(10)	1.478(3)	O(1)-Sm(1)-N(1)	63.92(5)
N(3)-C(9)	1.480(3)	O(5)-Sm(1)-N(3)	73.50(5)
N(4)-C(18)	1.478(2)	O(7)-Sm(1)-N(3)	131.63(5)
N(4)-C(12)	1.479(3)	O(3)-Sm(1)-N(3)	77.99(5)
N(4)-C(11)	1.496(2)	O(1)-Sm(1)-N(3)	141.36(5)
N(5)-C(14)	1.471(3)	N(1)-Sm(1)-N(3)	130.02(5)
N(5)-C(13)	1.479(3)	O(5)-Sm(1)-N(5)	130.71(5)
C(1)-C(2)	1.388(3)	O(7)-Sm(1)-N(5)	73.21(5)
C(1)-C(6)	1.520(3)	O(3)-Sm(1)-N(5)	139.83(5)
C(2)-C(3)	1.390(4)	O(1)-Sm(1)-N(5)	78.41(5)
C(3)-C(4)	1.383(4)	N(1)-Sm(1)-N(5)	130.45(5)
C(4)-C(5)	1.394(3)	N(3)-Sm(1)-N(5)	99.53(6)
C(5)-C(7)	1.521(3)	O(5)-Sm(1)-N(4)	139.46(5)
C(8)-C(9)	1.518(3)	O(7)-Sm(1)-N(4)	65.67(5)
C(10)-C(11)	1.514(3)	O(3)-Sm(1)-N(4)	74.43(5)
C(12)-C(13)	1.520(3)	O(1)-Sm(1)-N(4)	140.31(5)
C(14)-C(15)	1.510(3)	N(1)-Sm(1)-N(4)	125.36(5)
C(16)-C(17)	1.524(3)	N(3)-Sm(1)-N(4)	67.40(5)
C(18)-C(19)	1.535(3)	N(5)-Sm(1)-N(4)	67.97(5)
N(6)-C(33)	1.515(3)	O(5)-Sm(1)-N(2)	65.63(5)
N(6)-C(25)	1.520(3)	O(7)-Sm(1)-N(2)	138.22(5)
N(6)-C(29)	1.525(2)	O(3)-Sm(1)-N(2)	141.29(5)
N(6)-C(21)	1.526(3)	O(1)-Sm(1)-N(2)	76.02(5)
C(21)-C(22)	1.525(3)	N(1)-Sm(1)-N(2)	127.15(5)
C(22)-C(23)	1.528(3)	N(3)-Sm(1)-N(2)	68.16(5)
C(23)-C(24)	1.527(3)	N(5)-Sm(1)-N(2)	66.65(5)
C(25)-C(26)	1.525(3)	N(4)-Sm(1)-N(2)	107.50(5)
C(26)-C(27)	1.500(4)	C(7)-O(1)-Sm(1)	125.37(12)

C(6)-O(3)-Sm(1)	125.80(13)	O(8)-C(19)-O(7)	124.58(18)
C(17)-O(5)-Sm(1)	123.51(12)	O(8)-C(19)-C(18)	117.34(17)
C(19)-O(7)-Sm(1)	122.58(12)	O(7)-C(19)-C(18)	118.04(16)
C(5)-N(1)-C(1)	120.23(18)	C(33)-N(6)-C(25)	106.57(15)
C(5)-N(1)-Sm(1)	119.83(13)	C(33)-N(6)-C(29)	110.88(16)
C(1)-N(1)-Sm(1)	119.94(14)	C(25)-N(6)-C(29)	111.22(17)
C(16)-N(2)-C(8)	110.79(16)	C(33)-N(6)-C(21)	110.78(16)
C(16)-N(2)-C(15)	110.31(16)	C(25)-N(6)-C(21)	110.93(15)
C(8)-N(2)-C(15)	109.60(16)	C(29)-N(6)-C(21)	106.52(15)
C(16)-N(2)-Sm(1)	106.24(11)	C(22)-C(21)-N(6)	115.50(16)
C(8)-N(2)-Sm(1)	110.25(12)	C(21)-C(22)-C(23)	110.31(17)
C(15)-N(2)-Sm(1)	109.61(12)	C(24)-C(23)-C(22)	112.01(19)
C(10)-N(3)-C(9)	112.65(16)	N(6)-C(25)-C(26)	115.47(17)
C(10)-N(3)-Sm(1)	115.52(12)	C(27)-C(26)-C(25)	111.2(2)
C(9)-N(3)-Sm(1)	112.28(12)	C(26)-C(27)-C(28)	115.2(3)
C(18)-N(4)-C(12)	111.29(15)	C(30)-C(29)-N(6)	115.09(16)
C(18)-N(4)-C(11)	109.58(16)	C(31)-C(30)-C(29)	109.71(19)
C(12)-N(4)-C(11)	110.19(16)	C(30)-C(31)-C(32)	111.8(2)
C(18)-N(4)-Sm(1)	106.28(11)	N(6)-C(33)-C(34)	115.39(16)
C(12)-N(4)-Sm(1)	110.77(11)	C(35)-C(34)-C(33)	110.02(19)
C(11)-N(4)-Sm(1)	108.61(11)	C(34)-C(35)-C(36)	112.0(2)
C(14)-N(5)-C(13)	112.67(16)	O(51)-C(52)-C(53)	119.1(7)
C(14)-N(5)-Sm(1)	115.96(12)	O(51)-C(52)-C(51)	122.9(6)
C(13)-N(5)-Sm(1)	111.86(12)	C(53)-C(52)-C(51)	117.1(5)
N(1)-C(1)-C(2)	121.4(2)	O(61)-C(62)-C(64)	141.9(12)
N(1)-C(1)-C(6)	114.61(18)		
C(2)-C(1)-C(6)	124.01(19)		
C(1)-C(2)-C(3)	118.6(2)		
C(4)-C(3)-C(2)	119.7(2)		
C(3)-C(4)-C(5)	118.2(2)		
N(1)-C(5)-C(4)	121.9(2)		
N(1)-C(5)-C(7)	114.61(17)		
C(4)-C(5)-C(7)	123.52(19)		
O(4)-C(6)-O(3)	126.9(2)		
O(4)-C(6)-C(1)	117.9(2)		
O(3)-C(6)-C(1)	115.16(17)		
O(2)-C(7)-O(1)	126.8(2)		
O(2)-C(7)-C(5)	117.38(19)		
O(1)-C(7)-C(5)	115.76(17)		
N(2)-C(8)-C(9)	113.21(16)		
N(3)-C(9)-C(8)	111.14(16)		
N(3)-C(10)-C(11)	110.27(17)		
N(4)-C(11)-C(10)	112.00(17)		
N(4)-C(12)-C(13)	112.89(16)		
N(5)-C(13)-C(12)	111.03(16)		
N(5)-C(14)-C(15)	110.27(16)		
N(2)-C(15)-C(14)	112.50(17)		
N(2)-C(16)-C(17)	114.02(15)		
O(6)-C(17)-O(5)	123.96(18)		
O(6)-C(17)-C(16)	117.90(16)		
O(5)-C(17)-C(16)	118.09(16)		
N(4)-C(18)-C(19)	113.99(15)		

Table 4. Anisotropic displacement parameters ($Å^2 x 10^4$) for MLC07 (CCDC 655647). The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Sm(1)	103(1)	144(1)	102(1)	13(1)	7(1)	10(1)
O(1)	192(7)	191(7)	118(6)	-5(5)	-8(5)	27(5)
O(2)	562(12)	283(9)	148(7)	-69(6)	-67(7)	32(8)
O(3)	183(7)	195(7)	153(6)	48(5)	-15(5)	-1(5)
O(4)	324(9)	308(9)	274(9)	157(7)	-61(7)	-12(7)
O(5)	119(6)	222(7)	136(6)	36(5)	23(4)	20(5)
O(6)	234(7)	254(8)	157(6)	-9(5)	51(5)	31(5)
O(7)	159(6)	207(7)	115(6)	2(5)	12(5)	-22(5)
O(8)	175(6)	190(7)	153(6)	28(5)	21(5)	-7(5)
N(1)	122(6)	178(8)	155(6)	21(5)	33(5)	7(5)
N(2)	170(7)	169(8)	163(7)	32(6)	14(6)	15(5)
N(3)	139(7)	184(8)	184(8)	14(6)	-8(6)	-1(5)
N(4)	144(7)	190(8)	144(7)	-17(5)	17(5)	-25(5)
N(5)	147(7)	190(8)	147(7)	-1(6)	-5(5)	23(5)
C(1)	159(8)	196(10)	244(10)	61(7)	-2(7)	3(7)
C(2)	335(13)	191(11)	410(14)	79(9)	-75(11)	7(9)
C(3)	450(16)	181(12)	507(17)	-26(10)	-113(13)	23(10)
C(4)	274(11)	208(11)	352(12)	-73(9)	-24(9)	19(8)
C(5)	158(8)	183(9)	168(9)	-21(6)	25(6)	6(6)
C(6)	130(8)	254(10)	199(9)	88(7)	-13(7)	-7(7)
C(7)	199(9)	238(10)	115(8)	-22(6)	20(6)	19(7)
C(8)	233(10)	182(10)	229(10)	53(7)	11(7)	-29(7)
C(9)	155(8)	220(10)	225(10)	14(7)	15(7)	-26(7)
C(10)	174(9)	224(10)	224(10)	-37(7)	7(7)	-48(7)
C(11)	173(9)	243(10)	192(9)	-42(7)	-7(7)	-27(7)
C(12)	197(9)	204(10)	184(9)	-41(7)	14(7)	25(7)
C(13)	159(8)	212(10)	203(9)	-1(7)	28(7)	44(6)
C(14)	213(9)	192(10)	223(10)	38(7)	27(7)	55(7)
C(15)	213(9)	212(10)	191(9)	64(7)	5(7)	54(7)
C(16)	200(9)	229(10)	141(8)	48(7)	30(7)	31(7)
C(17)	119(7)	182(9)	153(7)	7(6)	-10(5)	-18(6)
C(18)	187(8)	231(10)	133(8)	5(6)	22(6)	-33(6)
C(19)	118(7)	172(9)	143(8)	14(6)	6(6)	17(6)
N(6)	155(7)	205(8)	176(8)	-71(6)	-12(6)	14(6)
C(21)	163(8)	248(10)	199(9)	-90(7)	0(7)	18(7)
C(22)	175(9)	343(12)	214(10)	-116(8)	28(7)	-27(8)
C(23)	231(10)	283(11)	246(11)	-49(8)	45(8)	-25(8)
C(24)	255(12)	362(14)	408(15)	-76(11)	93(10)	-52(9)
C(25)	191(9)	222(10)	189(9)	-54(7)	-16(7)	-13(7)
C(26)	378(13)	252(12)	289(12)	-35(9)	-3(10)	59(9)
C(27)	640(20)	279(14)	323(14)	14(10)	-125(13)	-28(12)
C(28)	670(20)	417(19)	620(20)	169(16)	-109(19)	81(16)
C(29)	173(9)	257(10)	220(9)	-133(7)	-38(7)	8(7)
C(30)	161(9)	328(13)	302(12)	-164(9)	-10(8)	-7(8)
C(31)	179(11)	640(20)	610(20)	-414(16)	10(11)	-63(11)

C(32)	197(12)	760(20)	690(20)	-361(19)	6(13)	-73(13)
C(33)	197(9)	192(9)	195(9)	-81(7)	-22(7)	40(7)
C(34)	302(12)	277(12)	212(10)	-50(8)	-23(8)	41(9)
C(35)	298(11)	265(11)	255(11)	-22(8)	-14(8)	64(8)
C(36)	428(15)	309(13)	323(13)	14(9)	-17(11)	55(10)
O(21)	424(11)	434(11)	248(9)	42(7)	74(7)	-159(8)
O(22)	377(9)	219(8)	187(7)	-4(6)	71(6)	62(6)
O(51)	870(30)	210(15)	980(30)	113(16)	-310(20)	-21(15)
C(51)	450(30)	820(40)	730(40)	150(30)	-100(20)	-140(30)
C(52)	810(40)	138(17)	680(30)	6(17)	-250(30)	154(19)
C(53)	720(40)	550(30)	1230(60)	-480(40)	340(40)	-190(30)
O(61)	510(50)	440(40)	240(30)	-60(30)	-80(30)	220(30)
C(62)	580(90)	440(80)	860(110)	-220(70)	160(70)	-170(60)
C(64)	390(70)	560(90)	1800(200)	-830(120)	-310(100)	240(70)
O(71)	250(30)	350(40)	280(30)	-120(30)	50(20)	-40(20)

Table 5. Hydrogen bonds for MLC07 (CCDC 655647) [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(3)-H(3)...O(5)#1	0.93	2.15	3.010(2)	152.5
N(3)-H(3)...O(6)#1	0.93	2.53	3.288(2)	139.4
N(5)-H(5)...O(7)#2	0.93	2.18	3.030(2)	152.0
N(5)-H(5)...O(8)#2	0.93	2.47	3.261(2)	142.4
O(21)-H(21C)...O(4)#1	0.96	1.931(3)	2.889(2)	175.4(14)
O(21)-H(21D)...O(6)	0.96	1.884(4)	2.832(2)	169.0(18)
O(22)-H(22C)...O(2)#1	0.96	1.912(2)	2.870(2)	174.7(7)
O(22)-H(22D)...O(8)#3	0.96	1.834(4)	2.7858(18)	170.7(17)
O(61)-H(61)...O(71)	0.84	2.30	2.831(9)	121.5

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,-y+1,-z+1
#2 -x+2,-y+1,-z+1
#3 x-1,y,z

CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Date 23 January 2007

Crystal Structure Analysis of:

MLC05

(shown below)

For Investigator: Morgan Cable (818) 354-4348

Advisor: Adrian Ponce/H. B. Gray (818) 354-8196

Account Number: NASA.00002-1-JPL.000174

By Michael W. Day 116 Beckman ext. 2734

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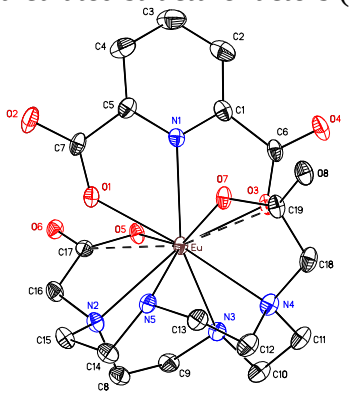
Contents



MLC05

Note: The crystallographic data have been deposited in the Cambridge Database (CCDC) and has been placed on hold pending further instructions from me. The deposition number is 634507. Ideally the CCDC would like the publication to contain a footnote of the type: "Crystallographic data have been deposited at the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK and copies can be obtained on request, free of charge, by quoting the publication citation and the deposition number 634507."

Table 1. Crystal data and structure refinement for MLC05 (CCDC 634507).

Empirical formula	$[C_{19}H_{25}N_5O_8Eu]^-[C_{16}H_{36}N]^+ \cdot 3(H_2O) \cdot 0.32(C_3H_6O) \cdot 0.68(C_3H_8O)$
Formula weight	957.23
Crystallization Solvent	Acetone/isopropanol/water
Crystal Habit	Prism
Crystal size	0.34 x 0.13 x 0.11 mm^3
Crystal color	Colorless

Data Collection

Type of diffractometer	Bruker SMART 1000
Wavelength	0.71073 Å MoKα
Data Collection Temperature	100(2) K
θ range for 25559 reflections used in lattice determination	2.18 to 37.61°
Unit cell dimensions	a = 13.1473(4) Å b = 13.2269(4) Å β= 90.0540(10)° c = 26.2248(8) Å
Volume	4560.4(2) $Å^3$
Z	4
Crystal system	Triclinic
Space group	P1
Density (calculated)	1.394 Mg/m^3
F(000)	2005
θ range for data collection	1.55 to 40.67°
Completeness to θ = 40.67°	93.9 %
Index ranges	$-23 \le h \le 23, -24 \le k \le 23, -47 \le l \le 43$
Data collection scan type	ω scans at 7 ϕ settings
Reflections collected	126185
Independent reflections	27668 [R_{int}= 0.1140]
Absorption coefficient	1.438 mm^{-1}
Absorption correction	None
Max. and min. transmission	0.8579 and 0.6407

Table 1 (cont.)

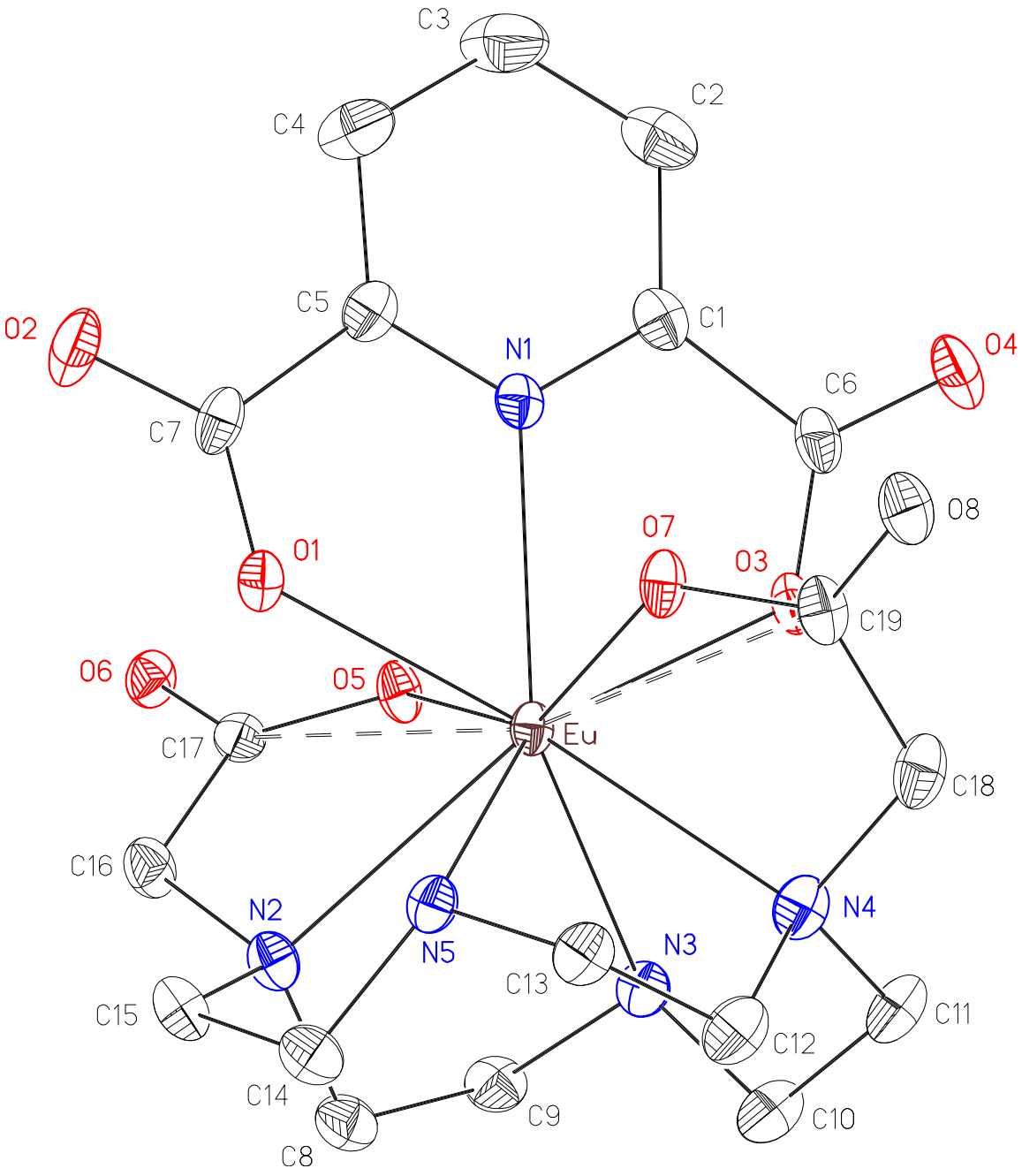
Structure solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 1990)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	SHELXL-97 (Sheldrick, 1997)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	27668 / 0 / 560
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	1.004
Final R indices [$I>2\sigma(I)$, 12878 reflections]	R1 = 0.0437, *w*R2 = 0.0750
R indices (all data)	R1 = 0.1187, *w*R2 = 0.0854
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.004
Average shift/error	0.000
Largest diff. peak and hole	4.742 and -2.402 e.Å^{-3}

Special Refinement Details

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.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C2
C5
C1
O2
N1
O4
C7
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O5
Eu
C17
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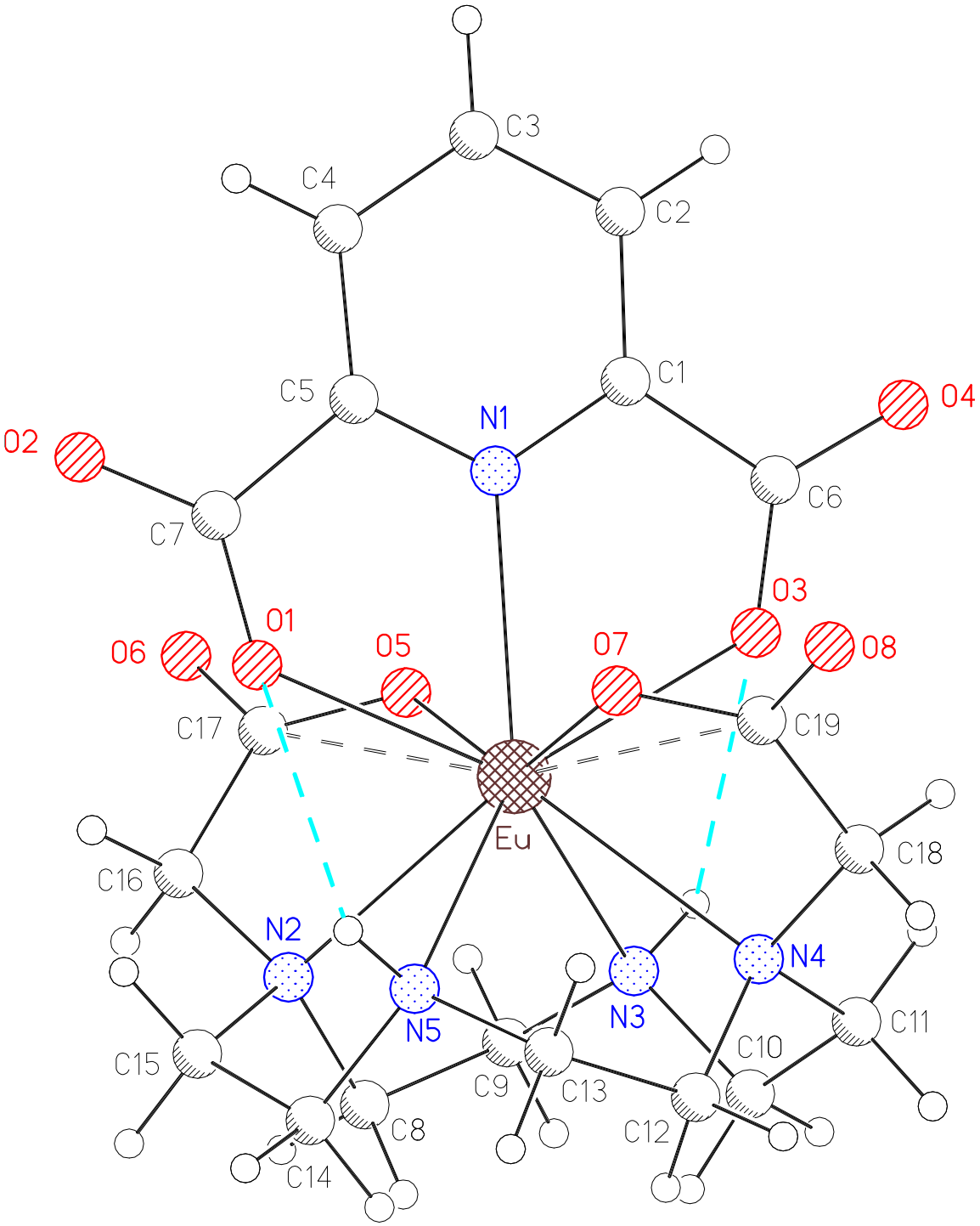
N4
C13
N3
C15
C12
C11
C14
C9
C10
C8

D19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C17
C19
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C11
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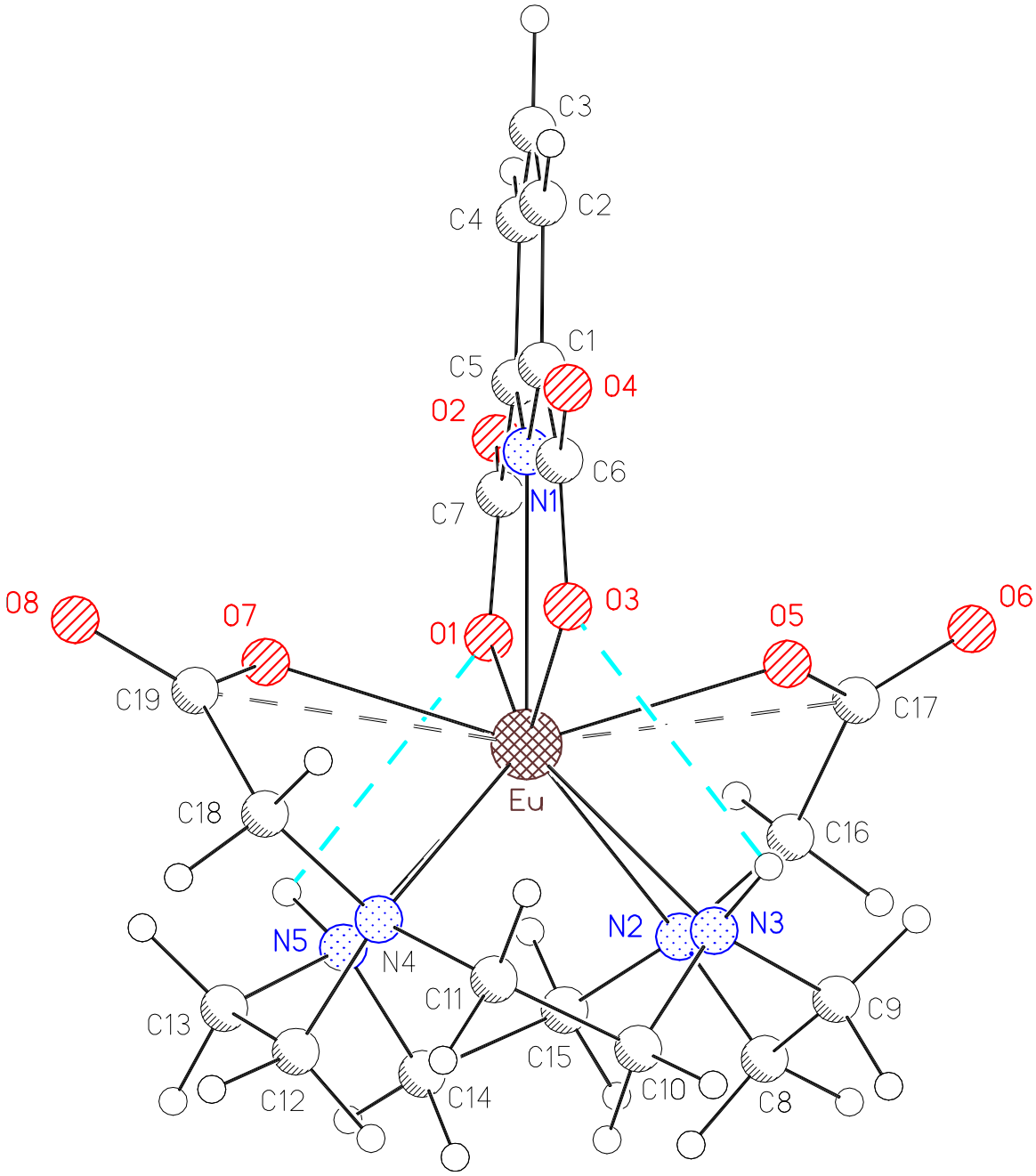
C10
C9
C13
C8
C12
C14

D20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C7
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O6
C19
C17
Eu
C18
C16
N5
N4
N2
N3
C13
C11
C15
C9
C12
C14
C10
C8

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for MLC05 (CCDC 634507). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}	Occ
Eu	2482(1)	42(1)	4988(1)	13(1)	1
O(1)	1700(1)	-916(1)	5660(1)	17(1)	1
O(2)	1405(1)	-2377(1)	6074(1)	29(1)	1
O(3)	3302(1)	-577(1)	4226(1)	18(1)	1
O(4)	3666(2)	-1811(2)	3671(1)	33(1)	1
O(5)	3952(1)	-588(1)	5414(1)	16(1)	1
O(6)	4674(1)	-1048(1)	6147(1)	17(1)	1
O(7)	1036(1)	-372(1)	4488(1)	18(1)	1
O(8)	273(1)	-413(1)	3728(1)	24(1)	1
N(1)	2504(1)	-1847(1)	4880(1)	14(1)	1
N(2)	2944(1)	1026(2)	5847(1)	17(1)	1
N(3)	3919(1)	1359(1)	4850(1)	17(1)	1
N(4)	2024(1)	1421(2)	4281(1)	19(1)	1
N(5)	1041(1)	1232(1)	5286(1)	17(1)	1
C(1)	2899(2)	-2254(2)	4459(1)	18(1)	1
C(2)	2928(2)	-3296(2)	4384(1)	29(1)	1
C(3)	2559(2)	-3920(2)	4766(1)	35(1)	1
C(4)	2161(2)	-3496(2)	5208(1)	29(1)	1
C(5)	2135(2)	-2437(2)	5250(1)	18(1)	1
C(6)	3319(2)	-1489(2)	4083(1)	18(1)	1
C(7)	1713(2)	-1876(2)	5707(1)	18(1)	1
C(8)	3637(2)	1886(2)	5742(1)	21(1)	1
C(9)	4419(2)	1659(2)	5333(1)	20(1)	1
C(10)	3657(2)	2237(2)	4530(1)	24(1)	1
C(11)	2969(2)	1910(2)	4095(1)	23(1)	1
C(12)	1323(2)	2197(2)	4492(1)	24(1)	1
C(13)	549(2)	1762(2)	4856(1)	21(1)	1
C(14)	1299(2)	1924(2)	5709(1)	21(1)	1
C(15)	1988(2)	1395(2)	6088(1)	20(1)	1
C(16)	3443(2)	272(2)	6180(1)	19(1)	1
C(17)	4078(2)	-509(2)	5893(1)	15(1)	1
C(18)	1527(2)	858(2)	3860(1)	23(1)	1
C(19)	887(1)	-34(2)	4037(1)	18(1)	1
N(6)	6382(1)	2794(1)	2785(1)	17(1)	1
C(20)	7191(2)	3434(2)	2522(1)	21(1)	1
C(21)	8262(2)	3295(2)	2731(1)	26(1)	1
C(22)	8999(2)	4001(3)	2463(1)	43(1)	1
C(23)	10103(2)	3786(3)	2615(1)	50(1)	1
C(24)	6676(2)	1685(2)	2785(1)	20(1)	1
C(25)	6818(2)	1216(2)	2260(1)	27(1)	1
C(26)	7080(2)	113(2)	2307(1)	29(1)	1
C(27)	7262(2)	-389(2)	1791(1)	38(1)	1
C(28)	5397(2)	2956(2)	2488(1)	20(1)	1
C(29)	4473(2)	2393(2)	2688(1)	24(1)	1
C(30)	3638(2)	2365(2)	2286(1)	27(1)	1
C(31)	2678(2)	1825(2)	2470(1)	37(1)	1

C(32)	6253(2)	3105(2)	3340(1)	18(1)	1
C(33)	6042(2)	4216(2)	3431(1)	26(1)	1
C(34)	5672(2)	4384(2)	3972(1)	30(1)	1
C(35)	5627(3)	5483(2)	4128(1)	51(1)	1
O(11)	5766(1)	4998(1)	1879(1)	27(1)	1
O(12)	9151(2)	977(2)	3130(1)	34(1)	1
O(51A)	1461(4)	4404(3)	6187(2)	56(2)	0.685(4)
C(52A)	546(4)	4383(4)	6230(2)	51(2)	0.685(4)
C(53A)	16(4)	3724(4)	6593(2)	51(1)	0.685(4)
C(54A)	-150(10)	4899(10)	5837(5)	137(6)	0.685(4)
O(50A)	-1088(18)	2273(18)	6469(9)	617(15)	0.685(4)
O(51B)	1254(8)	4293(6)	5372(4)	82(4)	0.315(4)
C(52B)	844(12)	4542(8)	5759(5)	61(4)	0.315(4)
C(53B)	-37(16)	5278(11)	5958(7)	39(3)	0.315(4)
C(54B)	1210(20)	4450(30)	6257(9)	240(20)	0.315(4)
O(50B)	-141(9)	3709(9)	7102(5)	101(4)	0.315(4)

Table 3. Selected bond lengths [Å] and angles [°] for MLC05 (CCDC 634507).

Eu-O(7)	2.3740(15)	O(5)-Eu-N(4)	138.38(6)
Eu-O(5)	2.3801(14)	O(1)-Eu-N(4)	140.64(5)
Eu-O(1)	2.4038(15)	O(3)-Eu-N(4)	75.97(6)
Eu-O(3)	2.4140(15)	N(1)-Eu-N(4)	127.07(6)
Eu-N(1)	2.5146(18)	N(5)-Eu-N(4)	68.22(6)
Eu-N(5)	2.5848(18)	N(3)-Eu-N(4)	66.98(6)
Eu-N(3)	2.5947(18)	O(7)-Eu-N(2)	139.46(6)
Eu-N(4)	2.6692(18)	O(5)-Eu-N(2)	65.89(5)
Eu-N(2)	2.6711(18)	O(1)-Eu-N(2)	74.65(6)
Eu-C(17)	3.249(2)	O(3)-Eu-N(2)	139.65(5)
Eu-C(19)	3.2578(18)	N(1)-Eu-N(2)	125.21(5)
		N(5)-Eu-N(2)	67.34(6)
O(7)-Eu-O(5)	145.85(6)	N(3)-Eu-N(2)	68.04(6)
O(7)-Eu-O(1)	86.57(5)	N(4)-Eu-N(2)	107.73(7)
O(5)-Eu-O(1)	79.59(5)	O(7)-Eu-C(17)	150.24(5)
O(7)-Eu-O(3)	79.77(5)	O(5)-Eu-C(17)	19.27(5)
O(5)-Eu-O(3)	84.64(5)	O(1)-Eu-C(17)	67.79(5)
O(1)-Eu-O(3)	128.30(6)	O(3)-Eu-C(17)	103.91(5)
O(7)-Eu-N(1)	73.56(5)	N(1)-Eu-C(17)	81.50(5)
O(5)-Eu-N(1)	72.30(5)	N(5)-Eu-C(17)	112.87(5)
O(1)-Eu-N(1)	64.12(5)	N(3)-Eu-C(17)	77.48(5)
O(3)-Eu-N(1)	64.18(5)	N(4)-Eu-C(17)	143.74(6)
O(7)-Eu-N(5)	73.75(6)	N(2)-Eu-C(17)	49.23(5)
O(5)-Eu-N(5)	131.78(5)	O(7)-Eu-C(19)	19.10(5)
O(1)-Eu-N(5)	77.57(6)	O(5)-Eu-C(19)	150.37(5)
O(3)-Eu-N(5)	141.69(5)	O(1)-Eu-C(19)	105.62(5)
N(1)-Eu-N(5)	130.32(6)	O(3)-Eu-C(19)	69.12(5)
O(7)-Eu-N(3)	131.38(6)	N(1)-Eu-C(19)	83.68(6)
O(5)-Eu-N(3)	73.13(6)	N(5)-Eu-C(19)	77.21(5)
O(1)-Eu-N(3)	140.21(5)	N(3)-Eu-C(19)	112.48(6)
O(3)-Eu-N(3)	77.68(6)	N(4)-Eu-C(19)	49.06(6)
N(1)-Eu-N(3)	130.00(6)	N(2)-Eu-C(19)	143.73(6)
N(5)-Eu-N(3)	99.67(6)	C(17)-Eu-C(19)	165.18(6)
O(7)-Eu-N(4)	66.01(6)		

Table 4. Bond lengths [Å] and angles [°] for MLC05 (CCDC 634507).

Eu-O(7)	2.3740(15)	C(24)-C(25)	1.521(3)
Eu-O(5)	2.3801(14)	C(25)-C(26)	1.504(3)
Eu-O(1)	2.4038(15)	C(26)-C(27)	1.528(4)
Eu-O(3)	2.4140(15)	C(28)-C(29)	1.520(3)
Eu-N(1)	2.5146(18)	C(29)-C(30)	1.523(3)
Eu-N(5)	2.5848(18)	C(30)-C(31)	1.529(4)
Eu-N(3)	2.5947(18)	C(32)-C(33)	1.514(3)
Eu-N(4)	2.6692(18)	C(33)-C(34)	1.518(3)
Eu-N(2)	2.6711(18)	C(34)-C(35)	1.511(4)
Eu-C(17)	3.249(2)	O(51A)-C(52A)	1.209(7)
Eu-C(19)	3.2578(18)	C(52A)-C(53A)	1.467(8)
O(1)-C(7)	1.275(3)	C(52A)-C(54A)	1.538(13)
O(2)-C(7)	1.238(3)	O(51B)-C(52B)	1.194(14)
O(3)-C(6)	1.264(3)	C(52B)-C(54B)	1.40(3)
O(4)-C(6)	1.247(3)	C(52B)-C(53B)	1.60(2)
O(5)-C(17)	1.273(2)		
O(6)-C(17)	1.251(2)	O(7)-Eu-O(5)	145.85(6)
O(7)-C(19)	1.278(2)	O(7)-Eu-O(1)	86.57(5)
O(8)-C(19)	1.249(2)	O(5)-Eu-O(1)	79.59(5)
N(1)-C(1)	1.334(3)	O(7)-Eu-O(3)	79.77(5)
N(1)-C(5)	1.337(3)	O(5)-Eu-O(3)	84.64(5)
N(2)-C(16)	1.477(3)	O(1)-Eu-O(3)	128.30(6)
N(2)-C(8)	1.484(3)	O(7)-Eu-N(1)	73.56(5)
N(2)-C(15)	1.490(3)	O(5)-Eu-N(1)	72.30(5)
N(3)-C(10)	1.473(3)	O(1)-Eu-N(1)	64.12(5)
N(3)-C(9)	1.481(3)	O(3)-Eu-N(1)	64.18(5)
N(4)-C(18)	1.482(3)	O(7)-Eu-N(5)	73.75(6)
N(4)-C(11)	1.484(3)	O(5)-Eu-N(5)	131.78(5)
N(4)-C(12)	1.486(3)	O(1)-Eu-N(5)	77.57(6)
N(5)-C(13)	1.477(3)	O(3)-Eu-N(5)	141.69(5)
N(5)-C(14)	1.477(3)	N(1)-Eu-N(5)	130.32(6)
C(1)-C(2)	1.393(3)	O(7)-Eu-N(3)	131.38(6)
C(1)-C(6)	1.518(3)	O(5)-Eu-N(3)	73.13(6)
C(2)-C(3)	1.388(4)	O(1)-Eu-N(3)	140.21(5)
C(3)-C(4)	1.391(4)	O(3)-Eu-N(3)	77.68(6)
C(4)-C(5)	1.405(3)	N(1)-Eu-N(3)	130.00(6)
C(5)-C(7)	1.515(3)	N(5)-Eu-N(3)	99.67(6)
C(8)-C(9)	1.516(3)	O(7)-Eu-N(4)	66.01(6)
C(10)-C(11)	1.518(3)	O(5)-Eu-N(4)	138.38(6)
C(12)-C(13)	1.511(3)	O(1)-Eu-N(4)	140.64(5)
C(14)-C(15)	1.516(3)	O(3)-Eu-N(4)	75.97(6)
C(16)-C(17)	1.526(3)	N(1)-Eu-N(4)	127.07(6)
C(18)-C(19)	1.521(3)	N(5)-Eu-N(4)	68.22(6)
N(6)-C(24)	1.517(3)	N(3)-Eu-N(4)	66.98(6)
N(6)-C(32)	1.522(3)	O(7)-Eu-N(2)	139.46(6)
N(6)-C(20)	1.524(3)	O(5)-Eu-N(2)	65.89(5)
N(6)-C(28)	1.525(3)	O(1)-Eu-N(2)	74.65(6)
C(20)-C(21)	1.522(3)	O(3)-Eu-N(2)	139.65(5)
C(21)-C(22)	1.519(3)	N(1)-Eu-N(2)	125.21(5)
C(22)-C(23)	1.531(4)	N(5)-Eu-N(2)	67.34(6)

N(3)-Eu-N(2)	68.04(6)	N(1)-C(5)-C(4)	121.1(2)
N(4)-Eu-N(2)	107.73(7)	N(1)-C(5)-C(7)	114.95(19)
O(7)-Eu-C(17)	150.24(5)	C(4)-C(5)-C(7)	123.9(2)
O(5)-Eu-C(17)	19.27(5)	O(4)-C(6)-O(3)	126.2(2)
O(1)-Eu-C(17)	67.79(5)	O(4)-C(6)-C(1)	117.9(2)
O(3)-Eu-C(17)	103.91(5)	O(3)-C(6)-C(1)	115.84(19)
N(1)-Eu-C(17)	81.50(5)	O(2)-C(7)-O(1)	127.1(2)
N(5)-Eu-C(17)	112.87(5)	O(2)-C(7)-C(5)	118.3(2)
N(3)-Eu-C(17)	77.48(5)	O(1)-C(7)-C(5)	114.62(19)
N(4)-Eu-C(17)	143.74(6)	N(2)-C(8)-C(9)	113.37(18)
N(2)-Eu-C(17)	49.23(5)	N(3)-C(9)-C(8)	110.88(17)
O(7)-Eu-C(19)	19.10(5)	N(3)-C(10)-C(11)	110.02(19)
O(5)-Eu-C(19)	150.37(5)	N(4)-C(11)-C(10)	112.06(19)
O(1)-Eu-C(19)	105.62(5)	N(4)-C(12)-C(13)	112.99(19)
O(3)-Eu-C(19)	69.12(5)	N(5)-C(13)-C(12)	111.68(18)
N(1)-Eu-C(19)	83.68(6)	N(5)-C(14)-C(15)	110.13(18)
N(5)-Eu-C(19)	77.21(5)	N(2)-C(15)-C(14)	112.10(18)
N(3)-Eu-C(19)	112.48(6)	N(2)-C(16)-C(17)	114.17(17)
N(4)-Eu-C(19)	49.06(6)	O(6)-C(17)-O(5)	124.1(2)
N(2)-Eu-C(19)	143.73(6)	O(6)-C(17)-C(16)	117.79(18)
C(17)-Eu-C(19)	165.18(6)	O(5)-C(17)-C(16)	118.07(18)
C(7)-O(1)-Eu	126.12(14)	O(6)-C(17)-Eu	157.04(15)
C(6)-O(3)-Eu	125.35(14)	O(5)-C(17)-Eu	38.08(10)
C(17)-O(5)-Eu	122.65(14)	C(16)-C(17)-Eu	81.64(11)
C(19)-O(7)-Eu	123.46(14)	N(4)-C(18)-C(19)	113.93(17)
C(1)-N(1)-C(5)	120.5(2)	O(8)-C(19)-O(7)	123.9(2)
C(1)-N(1)-Eu	119.92(14)	O(8)-C(19)-C(18)	117.99(18)
C(5)-N(1)-Eu	119.60(14)	O(7)-C(19)-C(18)	118.03(18)
C(16)-N(2)-C(8)	110.82(17)	O(8)-C(19)-Eu	157.66(18)
C(16)-N(2)-C(15)	110.20(17)	O(7)-C(19)-Eu	37.44(10)
C(8)-N(2)-C(15)	110.22(18)	C(18)-C(19)-Eu	81.62(11)
C(16)-N(2)-Eu	105.65(12)	C(24)-N(6)-C(32)	106.84(16)
C(8)-N(2)-Eu	110.79(13)	C(24)-N(6)-C(20)	111.10(17)
C(15)-N(2)-Eu	109.06(12)	C(32)-N(6)-C(20)	111.13(17)
C(10)-N(3)-C(9)	112.29(18)	C(24)-N(6)-C(28)	110.59(17)
C(10)-N(3)-Eu	115.98(13)	C(32)-N(6)-C(28)	110.74(17)
C(9)-N(3)-Eu	112.52(13)	C(20)-N(6)-C(28)	106.50(16)
C(18)-N(4)-C(11)	110.08(18)	C(21)-C(20)-N(6)	114.57(18)
C(18)-N(4)-C(12)	110.54(17)	C(22)-C(21)-C(20)	110.45(19)
C(11)-N(4)-C(12)	109.94(19)	C(21)-C(22)-C(23)	111.8(2)
C(18)-N(4)-Eu	105.84(14)	N(6)-C(24)-C(25)	115.20(18)
C(11)-N(4)-Eu	109.68(13)	C(26)-C(25)-C(24)	110.46(19)
C(12)-N(4)-Eu	110.70(13)	C(25)-C(26)-C(27)	112.6(2)
C(13)-N(5)-C(14)	112.27(18)	C(29)-C(28)-N(6)	115.72(17)
C(13)-N(5)-Eu	112.26(13)	C(28)-C(29)-C(30)	110.40(19)
C(14)-N(5)-Eu	115.85(13)	C(29)-C(30)-C(31)	112.7(2)
N(1)-C(1)-C(2)	121.8(2)	C(33)-C(32)-N(6)	115.71(18)
N(1)-C(1)-C(6)	114.27(19)	C(32)-C(33)-C(34)	110.40(19)
C(2)-C(1)-C(6)	123.9(2)	C(35)-C(34)-C(33)	114.0(2)
C(3)-C(2)-C(1)	118.5(2)	O(51A)-C(52A)-C(53A)	123.2(6)
C(2)-C(3)-C(4)	119.7(2)	O(51A)-C(52A)-C(54A)	121.2(8)
C(3)-C(4)-C(5)	118.4(2)	C(53A)-C(52A)-C(54A)	114.6(7)

O(51B)-C(52B)-C(54B)	128.0(19)
O(51B)-C(52B)-C(53B)	140.5(15)
C(54B)-C(52B)-C(53B)	89.6(15)

Table 5. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for MLC05 (CCDC 634507). The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Eu	106(1)	172(1)	111(1)	15(1)	-13(1)	1(1)
O(1)	175(7)	210(8)	129(7)	22(6)	18(5)	22(6)
O(2)	349(10)	306(10)	199(8)	116(7)	59(7)	42(8)
O(3)	198(8)	238(8)	101(7)	-8(6)	-5(5)	-16(6)
O(4)	485(12)	372(11)	142(8)	-82(8)	61(8)	-60(9)
O(5)	156(7)	230(8)	104(7)	-5(6)	-43(5)	24(6)
O(6)	167(7)	196(8)	143(7)	25(6)	-46(5)	-7(5)
O(7)	136(7)	265(8)	123(7)	43(6)	-36(5)	20(6)
O(8)	232(9)	323(9)	158(7)	-6(7)	-74(6)	18(7)
N(1)	113(7)	186(8)	128(8)	3(6)	-45(6)	-3(6)
N(2)	162(9)	210(9)	151(8)	-18(7)	-11(6)	16(7)
N(3)	148(8)	183(9)	174(9)	8(7)	8(7)	1(6)
N(4)	152(9)	230(10)	197(9)	45(8)	-15(7)	10(7)
N(5)	156(9)	187(9)	163(9)	27(7)	3(6)	12(6)
C(1)	119(9)	250(11)	165(10)	-29(8)	-31(7)	-1(8)
C(2)	267(13)	269(13)	325(14)	-106(11)	28(10)	-6(10)
C(3)	378(16)	209(12)	476(17)	-49(12)	75(13)	-13(11)
C(4)	281(13)	197(11)	381(15)	41(11)	48(11)	-12(9)
C(5)	143(10)	203(10)	191(10)	44(8)	-22(7)	19(7)
C(6)	149(10)	285(12)	115(9)	-14(8)	-26(7)	-26(8)
C(7)	129(10)	250(11)	163(10)	78(9)	-6(8)	10(8)
C(8)	210(11)	207(11)	214(11)	-34(9)	-33(8)	-10(8)
C(9)	153(10)	210(11)	226(11)	-8(9)	-41(8)	-50(8)
C(10)	209(11)	219(11)	282(13)	71(10)	-24(9)	-24(8)
C(11)	200(11)	250(12)	250(12)	107(10)	4(9)	-21(9)
C(12)	204(11)	243(12)	279(12)	76(10)	-23(9)	37(9)
C(13)	173(10)	216(11)	232(11)	30(9)	-20(8)	41(8)
C(14)	179(11)	220(11)	234(11)	-45(9)	-5(9)	30(8)
C(15)	170(10)	263(12)	172(10)	-56(9)	-6(8)	15(8)
C(16)	199(10)	221(11)	146(9)	-10(8)	-46(7)	24(7)
C(17)	130(9)	158(10)	158(9)	-4(8)	-18(7)	-24(7)
C(18)	177(11)	327(13)	179(10)	77(9)	-35(8)	-5(9)
C(19)	127(8)	276(11)	142(8)	2(10)	-29(6)	58(10)
N(6)	146(9)	216(9)	161(9)	64(7)	1(6)	1(7)
C(20)	160(10)	274(12)	209(11)	130(9)	19(8)	5(8)
C(21)	171(11)	323(13)	281(12)	135(10)	-15(9)	-8(9)
C(22)	203(13)	549(19)	524(19)	324(16)	-9(12)	-61(12)
C(23)	242(15)	640(20)	630(20)	269(18)	-3(14)	-85(14)
C(24)	167(10)	213(11)	207(11)	67(8)	3(8)	31(8)
C(25)	306(13)	302(14)	212(12)	36(10)	26(9)	49(10)
C(26)	301(12)	280(13)	290(11)	-3(11)	-28(9)	55(10)
C(27)	374(16)	411(16)	340(15)	-59(13)	4(12)	76(12)
C(28)	168(10)	265(12)	160(10)	81(9)	-24(8)	28(8)
C(29)	176(11)	348(13)	194(11)	90(10)	-23(8)	-19(9)
C(30)	268(13)	292(13)	257(13)	45(10)	-74(10)	-22(10)
C(31)	248(14)	390(16)	466(18)	69(13)	-109(12)	-69(11)

C(32)	180(10)	196(10)	158(9)	57(8)	9(7)	2(7)
C(33)	290(13)	201(11)	286(13)	65(10)	6(10)	-8(9)
C(34)	381(15)	237(13)	282(14)	-21(10)	34(11)	-6(10)
C(35)	610(20)	358(18)	560(20)	-86(16)	160(18)	-3(16)
O(11)	364(9)	263(9)	192(7)	30(8)	-82(6)	65(9)
O(12)	371(11)	419(12)	223(9)	24(8)	-77(8)	153(9)
O(51A)	224(18)	490(20)	960(40)	-60(20)	30(20)	-8(15)
C(52A)	280(20)	560(30)	700(40)	-270(30)	30(20)	60(20)
C(53A)	400(30)	540(30)	600(30)	-110(30)	20(20)	0(20)
C(54A)	560(60)	2450(180)	1110(100)	250(100)	110(70)	440(100)
O(51B)	1270(90)	350(50)	830(70)	-30(40)	480(60)	-110(50)
C(52B)	1050(120)	210(50)	570(80)	40(50)	360(80)	-120(50)
C(53B)	370(70)	420(60)	380(60)	80(50)	30(50)	80(40)
C(54B)	1400(300)	5200(600)	640(130)	-1500(200)	-210(160)	1100(300)

Table 6. Hydrogen bonds for MLC05 (CCDC 634507) [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(3)-H(3)...O(5)#1	0.93	2.23	3.059(2)	148.5
N(3)-H(3)...O(6)#1	0.93	2.45	3.232(2)	141.6
N(5)-H(5)...O(7)#2	0.93	2.17	3.016(2)	151.4
N(5)-H(5)...O(8)#2	0.93	2.54	3.294(3)	138.3
O(11)-H(11A)...O(4)#3	1.00	1.90	2.892(3)	175.7
O(11)-H(11B)...O(6)#4	1.00	1.80	2.769(2)	162.2
O(12)-H(12A)...O(2)#1	1.00	1.91	2.886(3)	164.7
O(12)-H(12B)...O(8)#5	1.00	1.88	2.830(2)	158.3
N(3)-H(3)...O(3)	0.93	2.82	3.144(2)	102.1
N(5)-H(5)...O(1)	0.93	2.81	3.128(2)	101.1

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

CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Date 1 September 2009

Crystal Structure Analysis of:

MLC13

(shown below)

For Investigator: Morgan Cable (818) 354-2539

Advisor: H. B. Gray ext. 6500

Account Number: AP1.HSARPA3-1-HSARPA.PONCE

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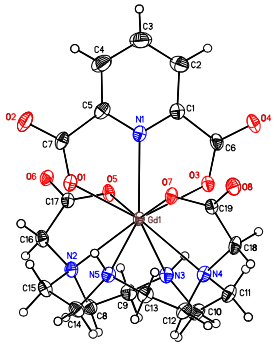
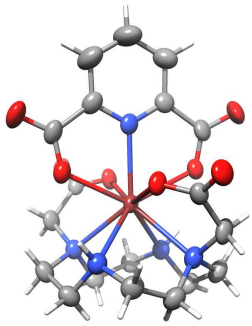
Contents



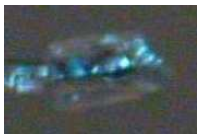


MLC13

Note: The crystallographic data have been deposited in the Cambridge Database (CCDC) and has been placed on hold pending further instructions from me. The deposition number is 746157. Ideally the CCDC would like the publication to contain a footnote of the type: "Crystallographic data have been deposited at the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK and copies can be obtained on request, free of charge, by quoting the publication citation and the deposition number 746157."

Table 1. Crystal data and structure refinement for MLC13 (CCDC 746175).

Empirical formula	$[C_{19}H_{25}N_5O_8Gd]^-$ $[C_{16}H_{36}N]+ \bullet 2(H_2O)$ $0.62(C_3H_6O)$ $0.38(C_2H_6O \bullet O)$
Formula weight	946.79
Crystallization Solvent	Ethanol/
Crystal Habit	Block
Crystal size	0.22 x 0.16 x 0.13 mm^3
Crystal color	Colorless



Data Collection

Type of diffractometer	Bruker KAPPA APEX II
Wavelength	0.71073 Å MoKα
Data Collection Temperature	100(2) K
θ range for 9360 reflections used in lattice determination	2.18 to 47.36°
Unit cell dimensions	a = 13.1910(5) Å b = 13.4544(5) Å β= 90.368(2)° c = 26.1712(9) Å
Volume	4644.7(3) $Å^3$
Z	4
Crystal system	Monoclinic
Space group	$P2_1/c$
Density (calculated)	1.354 Mg/m^3
F(000)	1975
θ range for data collection	2.16 to 47.72°
Completeness to θ = 47.72°	90.4 %
Index ranges	$-20 \le h \le 27$, $-27 \le k \le 27$, $-53 \le l \le 54$
Data collection scan type	ω scans; 19 settings
Reflections collected	245931
Independent reflections	39658 [R_{int}= 0.0563]
Absorption coefficient	1.487 mm^{-1}
Absorption correction	None
Max. and min. transmission	0.8302 and 0.7356

Table 1 (cont.)

Structure solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 2008)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Difference Fourier map
Structure refinement program	SHELXL-97 (Sheldrick, 2008)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	39658 / 0 / 664
Treatment of hydrogen atoms	Mixed
Goodness-of-fit on F^2	1.384
Final R indices [$I>2\sigma(I)$, 22019 reflections]	R1 = 0.0302, *w*R2 = 0.0482
R indices (all data)	R1 = 0.0805, *w*R2 = 0.0509
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.005
Average shift/error	0.000
Largest diff. peak and hole	3.917 and -2.616 e.Å^{-3}

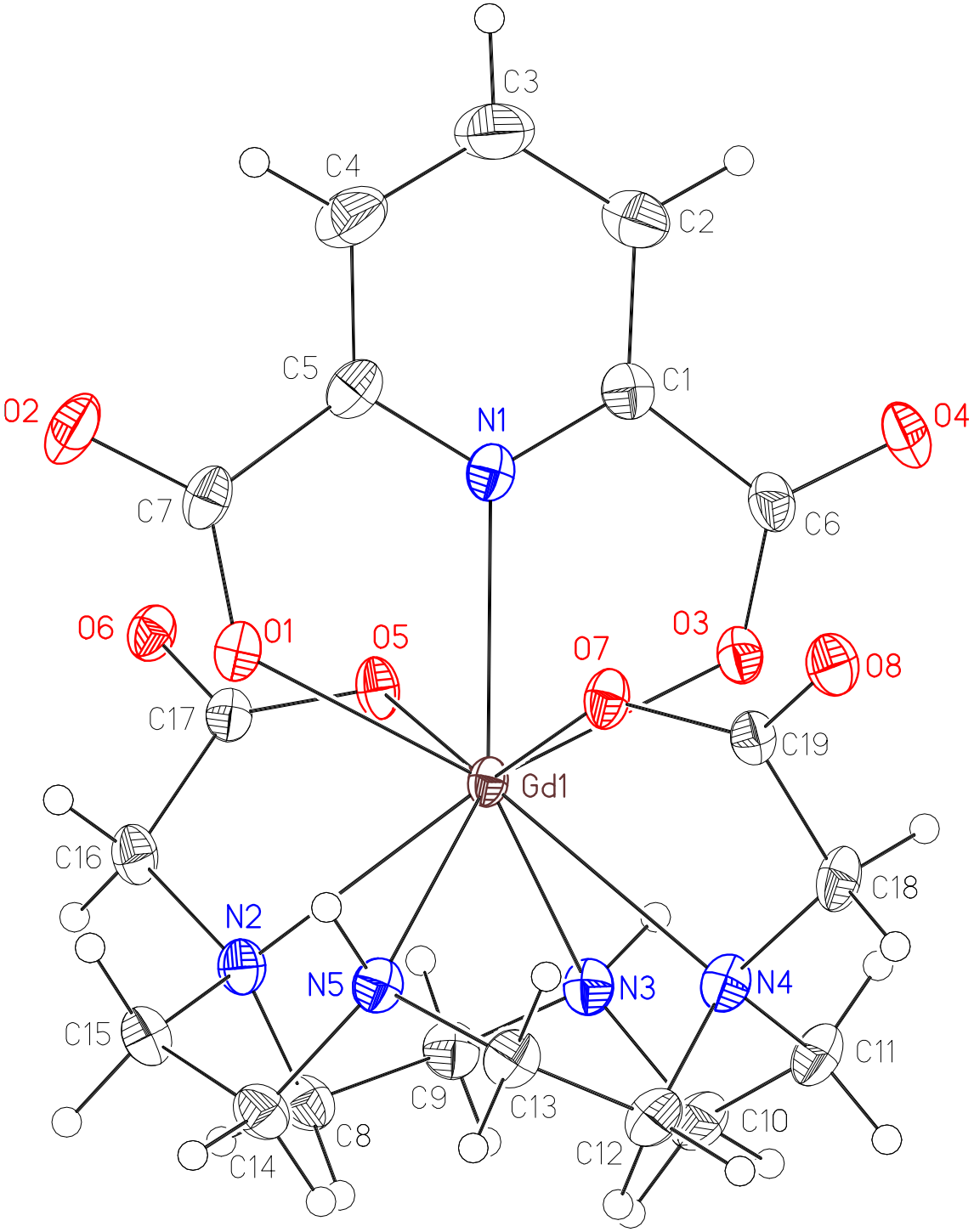
Special Refinement Details

Crystals were mounted on a glass fiber using Paratone oil then placed on the diffractometer under a nitrogen stream at 100K.

Hydrogen atoms in the Gadolinium dipicolinate macrocycle were refined without restraint. Hydrogen atoms of the tetramethyammonium counterion were restrained. The solvent region is disordered, containing acetone (62%) and ethanol/water (38%). These were refined without restraint (except for riding hydrogens). Additionally, the solvent region contains two ordered water molecules which were refined with no restraint on corresponding hydrogens.

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.


C3
C4
C2
C5
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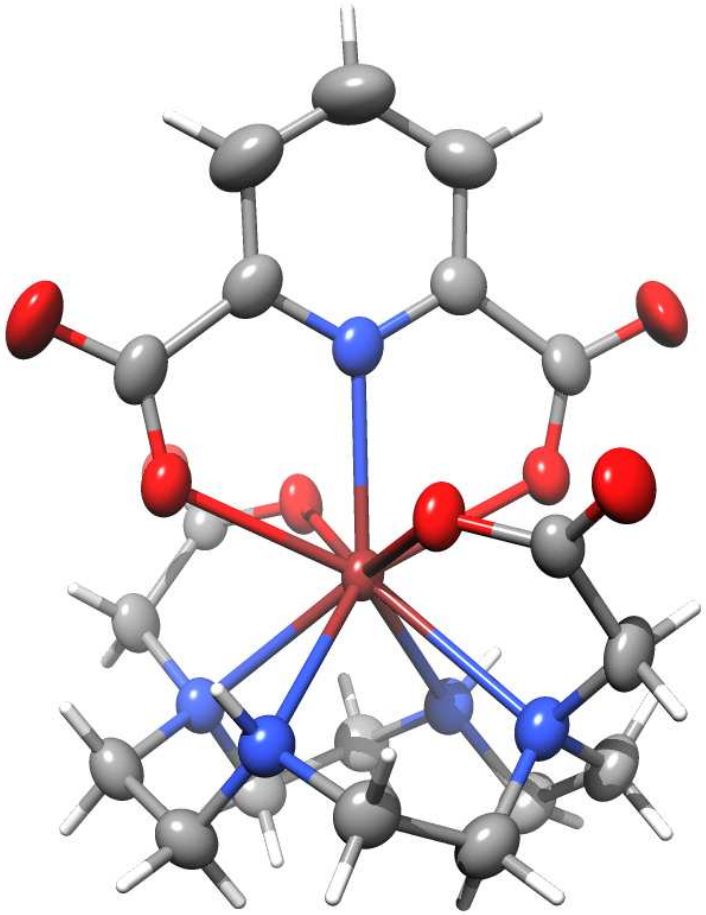
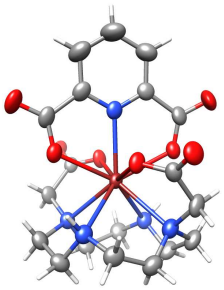
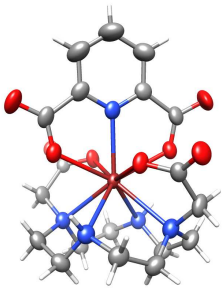
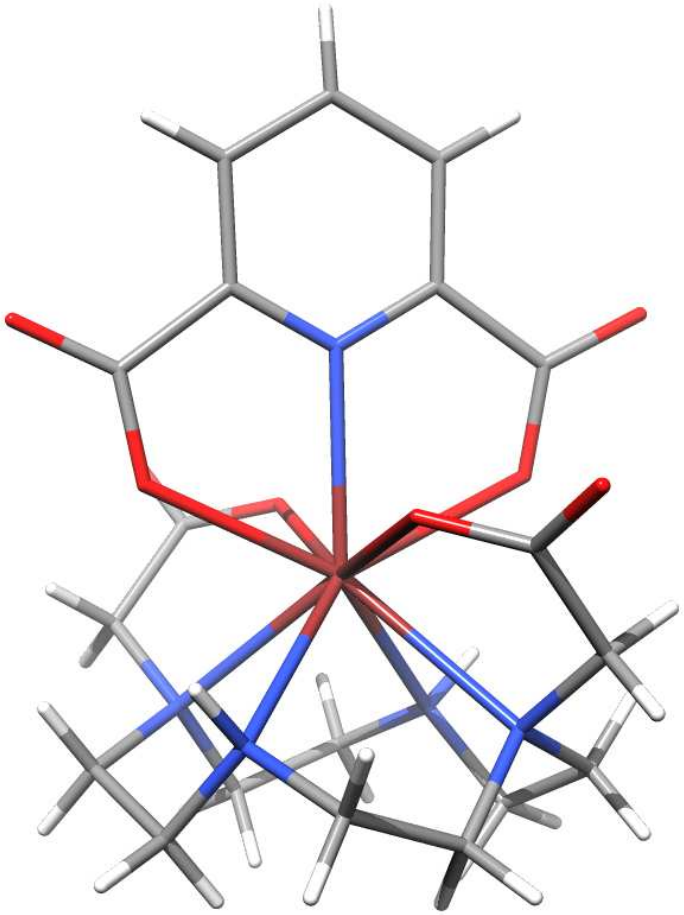
O4
C7
C6
O6
O1
O5
O7
O3
O8
C17
C19
Gd1
C16
C18
N2
N5
N3
N4
C15
C11
C9
C13
C8
C10
C14
C12

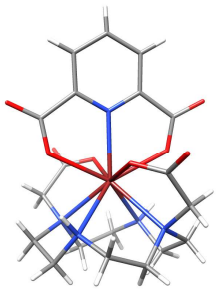


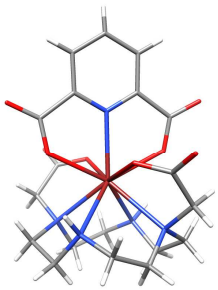


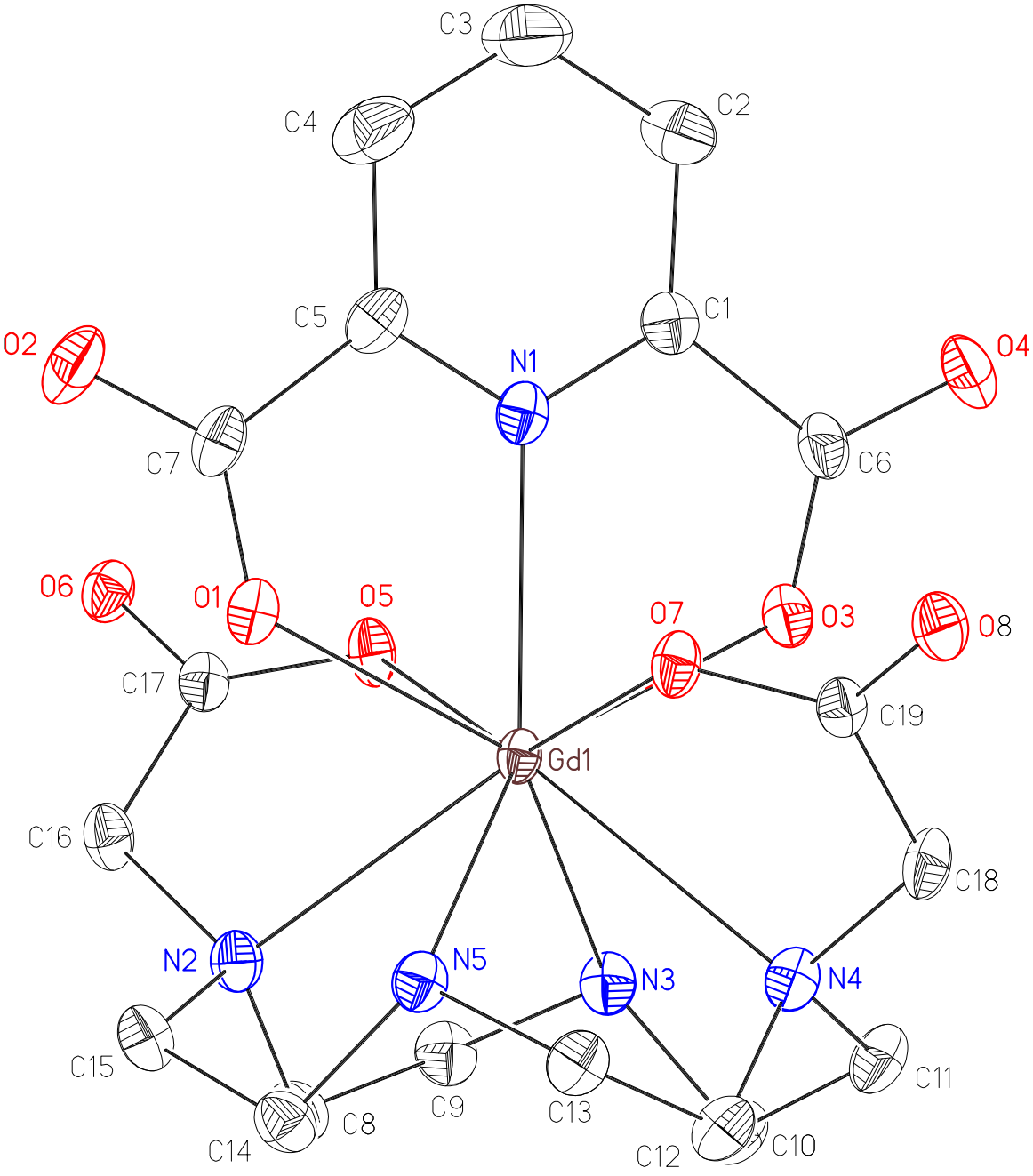


D35





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C17
C19
Gd1
C16
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N3
N4
C15
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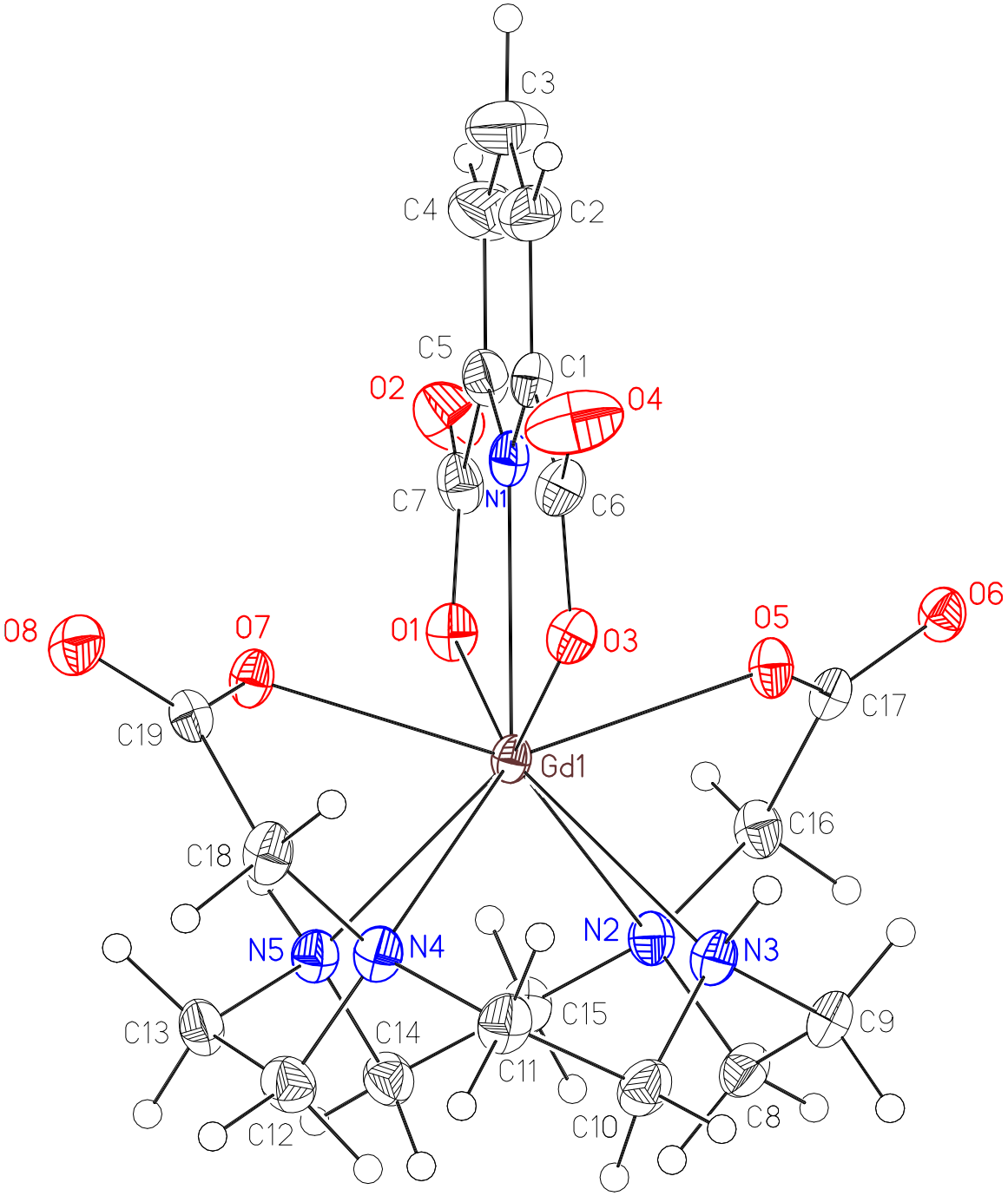
C13
C14
C8
C12
C10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C19
C17
Gd1
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N3
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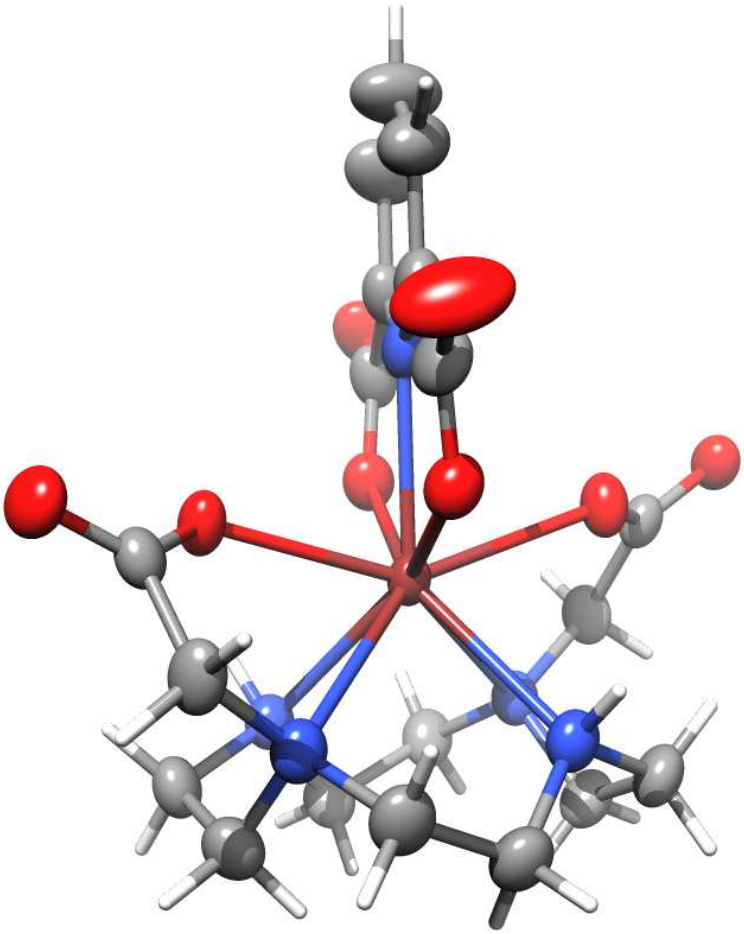
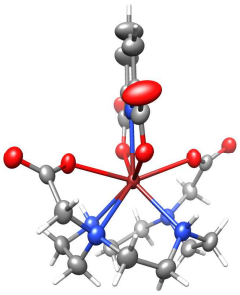
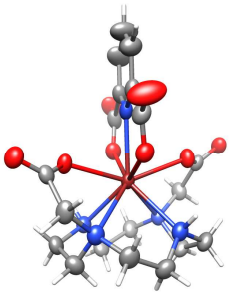
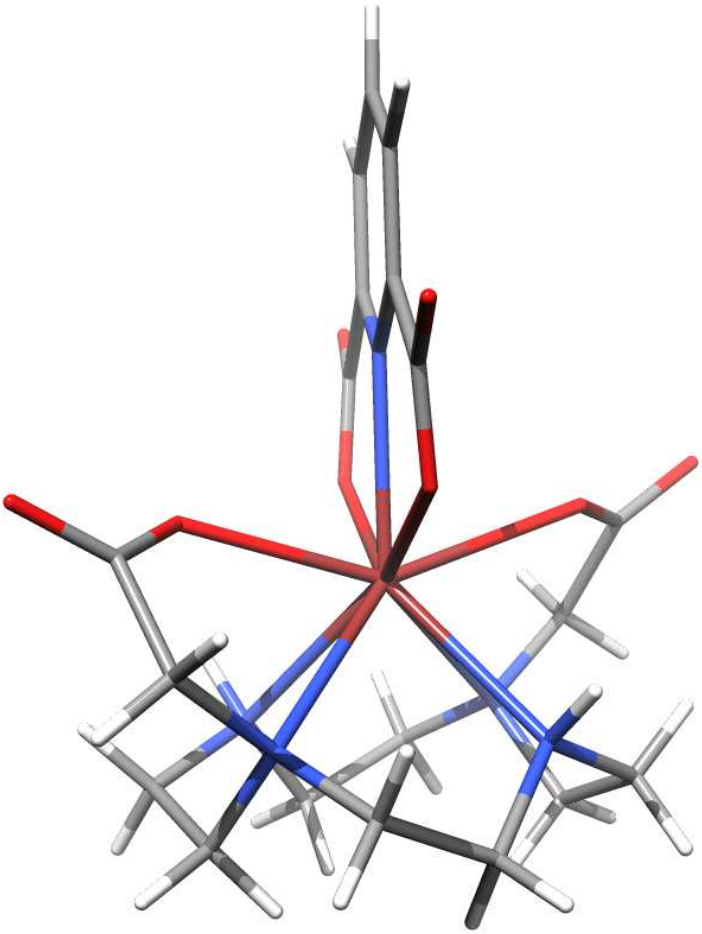
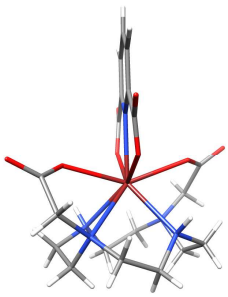
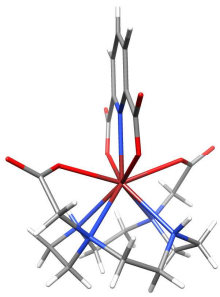




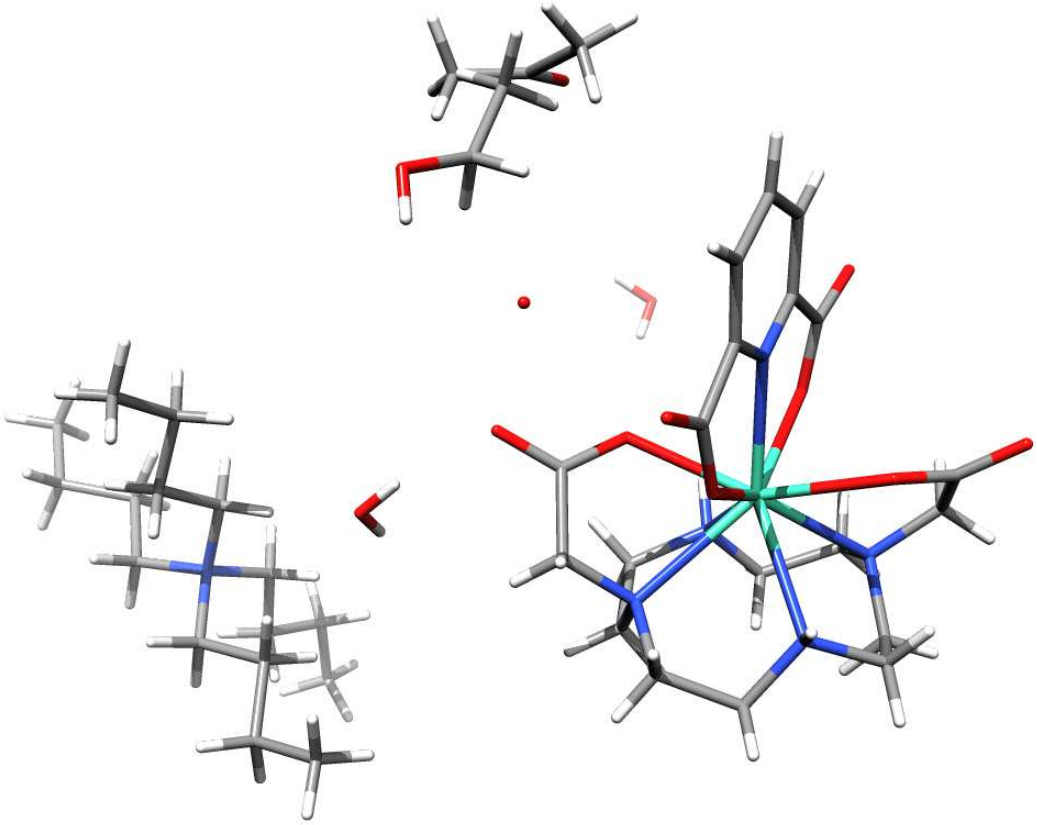








D40



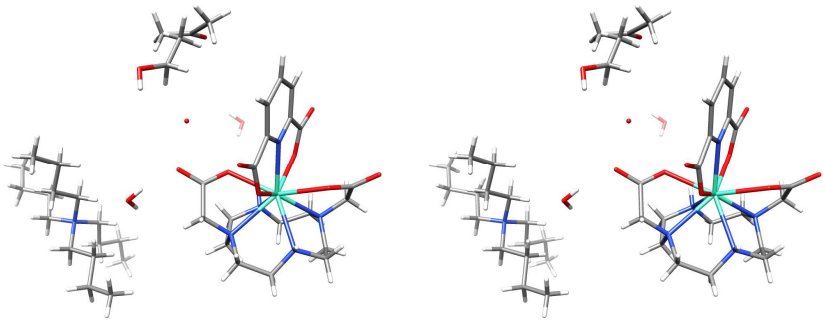


Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for MLC13 (CCDC 746175). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}	Occ
Gd(1)	7490(1)	4917(1)	5001(1)	13(1)	1
O(1)	6703(1)	5881(1)	4333(1)	19(1)	1
O(2)	6426(1)	7336(1)	3928(1)	30(1)	1
O(3)	8308(1)	5488(1)	5772(1)	19(1)	1
O(4)	8622(1)	6681(1)	6351(1)	39(1)	1
O(5)	8963(1)	5567(1)	4588(1)	18(1)	1
O(6)	9675(1)	6070(1)	3858(1)	18(1)	1
O(7)	6048(1)	5315(1)	5506(1)	18(1)	1
O(8)	5318(1)	5347(1)	6272(1)	24(1)	1
N(1)	7506(1)	6765(1)	5128(1)	17(1)	1
N(2)	7951(1)	3999(1)	4132(1)	17(1)	1
N(3)	8929(1)	3623(1)	5129(1)	18(1)	1
N(4)	7027(1)	3534(1)	5694(1)	19(1)	1
N(5)	6042(1)	3778(1)	4685(1)	18(1)	1
C(1)	7888(1)	7148(1)	5558(1)	18(1)	1
C(2)	7904(1)	8170(1)	5648(1)	28(1)	1
C(3)	7526(1)	8799(1)	5271(1)	38(1)	1
C(4)	7138(1)	8394(1)	4823(1)	33(1)	1
C(5)	7135(1)	7365(1)	4765(1)	21(1)	1
C(6)	8304(1)	6379(1)	5931(1)	20(1)	1
C(7)	6719(1)	6823(1)	4297(1)	20(1)	1
C(8)	8640(1)	3142(1)	4229(1)	21(1)	1
C(9)	9428(1)	3350(1)	4642(1)	21(1)	1
C(10)	8664(1)	2739(1)	5438(1)	23(1)	1
C(11)	7978(1)	3041(1)	5879(1)	23(1)	1
C(12)	6325(1)	2782(1)	5468(1)	23(1)	1
C(13)	5541(1)	3237(1)	5108(1)	21(1)	1
C(14)	6303(1)	3111(1)	4256(1)	21(1)	1
C(15)	6993(1)	3653(1)	3879(1)	20(1)	1
C(16)	8461(1)	4754(1)	3807(1)	20(1)	1
C(17)	9089(1)	5519(1)	4108(1)	16(1)	1
C(18)	6526(1)	4068(1)	6121(1)	21(1)	1
C(19)	5905(1)	4973(1)	5955(1)	17(1)	1
N(6)	1372(1)	2141(1)	7192(1)	20(1)	1
C(21)	1673(1)	3238(1)	7202(1)	22(1)	1
C(22)	1837(1)	3680(1)	7729(1)	29(1)	1
C(23)	2069(1)	4787(1)	7687(1)	29(1)	1
C(24)	2293(1)	5256(1)	8209(1)	40(1)	1
C(25)	2189(1)	1507(1)	7452(1)	25(1)	1
C(26)	3270(1)	1652(1)	7251(1)	30(1)	1
C(27)	3999(1)	924(1)	7509(1)	58(1)	1
C(28)	5108(1)	1149(2)	7379(1)	59(1)	1
C(29)	1242(1)	1851(1)	6635(1)	22(1)	1
C(30)	972(1)	767(1)	6537(1)	34(1)	1
C(31)	695(1)	590(1)	5986(1)	41(1)	1

C(32)	537(2)	-481(1)	5839(1)	61(1)	1
C(33)	383(1)	1976(1)	7488(1)	23(1)	1
C(34)	-548(1)	2528(1)	7282(1)	27(1)	1
C(35)	-1407(1)	2536(1)	7676(1)	28(1)	1
C(36)	-2365(1)	3059(1)	7484(1)	37(1)	1
O(21)	755(1)	5071(1)	3099(1)	27(1)	1
O(22)	4180(1)	3991(1)	6880(1)	37(1)	1
O(51)	3644(2)	9315(1)	5378(1)	71(1)	0.617(2)
C(51)	5077(2)	10064(4)	5838(2)	103(2)	0.617(2)
C(52)	3994(3)	9518(2)	5800(1)	70(1)	0.617(2)
C(53)	3504(2)	9414(2)	6205(1)	45(1)	0.617(2)
O(61)	5123(2)	8723(2)	7094(1)	45(1)	0.383(2)
C(62)	4987(4)	8771(4)	6547(2)	75(2)	0.383(2)
C(64)	4438(4)	9473(4)	6157(3)	72(2)	0.383(2)
O(71)	6124(2)	7291(2)	6479(1)	25(1)	0.383(2)

Table 3. Selected bond lengths [Å] and angles [°] for MLC13 (CCDC 746175).

Gd(1)-O(7)	2.3850(7)	O(3)-Gd(1)-N(5)	141.62(3)
Gd(1)-O(5)	2.3941(7)	N(1)-Gd(1)-N(5)	129.53(3)
Gd(1)-O(1)	2.4069(7)	O(7)-Gd(1)-N(3)	131.59(3)
Gd(1)-O(3)	2.4080(8)	O(5)-Gd(1)-N(3)	73.05(3)
Gd(1)-N(1)	2.5081(8)	O(1)-Gd(1)-N(3)	140.09(3)
Gd(1)-N(5)	2.5816(9)	O(3)-Gd(1)-N(3)	77.43(3)
Gd(1)-N(3)	2.5960(9)	N(1)-Gd(1)-N(3)	129.98(3)
Gd(1)-N(2)	2.6607(9)	N(5)-Gd(1)-N(3)	100.48(3)
Gd(1)-N(4)	2.6727(9)	O(7)-Gd(1)-N(2)	139.96(3)
		O(5)-Gd(1)-N(2)	66.05(3)
O(7)-Gd(1)-O(5)	145.15(3)	O(1)-Gd(1)-N(2)	74.30(3)
O(7)-Gd(1)-O(1)	86.56(3)	O(3)-Gd(1)-N(2)	139.52(2)
O(5)-Gd(1)-O(1)	79.85(3)	N(1)-Gd(1)-N(2)	124.89(3)
O(7)-Gd(1)-O(3)	79.50(3)	N(5)-Gd(1)-N(2)	67.89(3)
O(5)-Gd(1)-O(3)	84.32(3)	N(3)-Gd(1)-N(2)	68.18(3)
O(1)-Gd(1)-O(3)	128.75(3)	O(7)-Gd(1)-N(4)	65.99(3)
O(7)-Gd(1)-N(1)	73.11(3)	O(5)-Gd(1)-N(4)	138.60(3)
O(5)-Gd(1)-N(1)	72.07(3)	O(1)-Gd(1)-N(4)	140.23(3)
O(1)-Gd(1)-N(1)	64.25(3)	O(3)-Gd(1)-N(4)	75.90(3)
O(3)-Gd(1)-N(1)	64.50(3)	N(1)-Gd(1)-N(4)	127.00(3)
O(7)-Gd(1)-N(5)	73.71(3)	N(5)-Gd(1)-N(4)	68.35(3)
O(5)-Gd(1)-N(5)	132.39(3)	N(3)-Gd(1)-N(4)	67.37(3)
O(1)-Gd(1)-N(5)	76.86(3)	N(2)-Gd(1)-N(4)	108.11(3)

Table 4. Bond lengths [Å] and angles [°] for MLC13 (CCDC 746175).

Gd(1)-O(7)	2.3850(7)	C(12)-C(13)	1.5229(16)
Gd(1)-O(5)	2.3941(7)	C(12)-H(12A)	1.004(14)
Gd(1)-O(1)	2.4069(7)	C(12)-H(12B)	1.000(13)
Gd(1)-O(3)	2.4080(8)	C(13)-H(13A)	0.921(15)
Gd(1)-N(1)	2.5081(8)	C(13)-H(13B)	0.989(13)
Gd(1)-N(5)	2.5816(9)	C(14)-C(15)	1.5308(16)
Gd(1)-N(3)	2.5960(9)	C(14)-H(14A)	0.966(13)
Gd(1)-N(2)	2.6607(9)	C(14)-H(14B)	0.951(13)
Gd(1)-N(4)	2.6727(9)	C(15)-H(15A)	1.004(14)
O(1)-C(7)	1.2711(13)	C(15)-H(15B)	0.928(13)
O(2)-C(7)	1.2473(13)	C(16)-C(17)	1.5346(15)
O(3)-C(6)	1.2698(13)	C(16)-H(16A)	0.927(14)
O(4)-C(6)	1.2435(14)	C(16)-H(16B)	0.981(13)
O(5)-C(17)	1.2710(12)	C(18)-C(19)	1.5296(14)
O(6)-C(17)	1.2572(12)	C(18)-H(18A)	1.012(15)
O(7)-C(19)	1.2768(12)	C(18)-H(18B)	0.962(15)
O(8)-C(19)	1.2452(12)	N(6)-C(29)	1.5182(15)
N(1)-C(1)	1.3345(14)	N(6)-C(21)	1.5282(14)
N(1)-C(5)	1.3360(13)	N(6)-C(25)	1.5307(13)
N(2)-C(16)	1.4874(13)	N(6)-C(33)	1.5370(13)
N(2)-C(8)	1.4893(14)	C(21)-C(22)	1.5161(17)
N(2)-C(15)	1.4965(14)	C(22)-C(23)	1.5233(17)
N(3)-C(10)	1.4822(14)	C(23)-C(24)	1.532(2)
N(3)-C(9)	1.4851(14)	C(25)-C(26)	1.5362(16)
N(3)-H(3)	0.956(15)	C(26)-C(27)	1.5265(17)
N(4)-C(18)	1.4878(14)	C(27)-C(28)	1.535(2)
N(4)-C(12)	1.4905(15)	C(29)-C(30)	1.5234(16)
N(4)-C(11)	1.4974(14)	C(30)-C(31)	1.504(2)
N(5)-C(14)	1.4805(14)	C(31)-C(32)	1.506(2)
N(5)-C(13)	1.4842(14)	C(33)-C(34)	1.5312(16)
N(5)-H(5)	0.911(14)	C(34)-C(35)	1.5366(16)
C(1)-C(2)	1.3940(15)	C(35)-C(36)	1.5282(17)
C(1)-C(6)	1.5218(15)	O(21)-H(21D)	0.846(18)
C(2)-C(3)	1.3895(19)	O(21)-H(21C)	0.793(18)
C(2)-H(2)	0.973(15)	O(22)-H(22C)	0.825(17)
C(3)-C(4)	1.389(2)	O(22)-H(22D)	0.836(19)
C(3)-H(3A)	0.921(16)	O(51)-C(52)	1.227(4)
C(4)-C(5)	1.3934(16)	C(51)-C(52)	1.609(5)
C(4)-H(4)	0.937(16)	C(52)-C(53)	1.252(5)
C(5)-C(7)	1.5245(17)	O(61)-C(62)	1.445(7)
C(8)-C(9)	1.5191(17)	C(62)-C(64)	1.566(9)
C(8)-H(8A)	0.912(14)		
C(8)-H(8B)	0.989(14)	O(7)-Gd(1)-O(5)	145.15(3)
C(9)-H(9A)	0.976(13)	O(7)-Gd(1)-O(1)	86.56(3)
C(9)-H(9B)	0.965(13)	O(5)-Gd(1)-O(1)	79.85(3)
C(10)-C(11)	1.5270(16)	O(7)-Gd(1)-O(3)	79.50(3)
C(10)-H(10A)	0.971(14)	O(5)-Gd(1)-O(3)	84.32(3)
C(10)-H(10B)	0.979(13)	O(1)-Gd(1)-O(3)	128.75(3)
C(11)-H(11A)	0.955(13)	O(7)-Gd(1)-N(1)	73.11(3)
C(11)-H(11B)	0.948(12)	O(5)-Gd(1)-N(1)	72.07(3)

O(1)-Gd(1)-N(1)	64.25(3)	C(11)-N(4)-Gd(1)	109.53(6)
O(3)-Gd(1)-N(1)	64.50(3)	C(14)-N(5)-C(13)	112.16(8)
O(7)-Gd(1)-N(5)	73.71(3)	C(14)-N(5)-Gd(1)	115.28(6)
O(5)-Gd(1)-N(5)	132.39(3)	C(13)-N(5)-Gd(1)	112.63(7)
O(1)-Gd(1)-N(5)	76.86(3)	C(14)-N(5)-H(5)	112.6(9)
O(3)-Gd(1)-N(5)	141.62(3)	C(13)-N(5)-H(5)	107.9(9)
N(1)-Gd(1)-N(5)	129.53(3)	Gd(1)-N(5)-H(5)	94.9(9)
O(7)-Gd(1)-N(3)	131.59(3)	N(1)-C(1)-C(2)	121.92(10)
O(5)-Gd(1)-N(3)	73.05(3)	N(1)-C(1)-C(6)	114.23(9)
O(1)-Gd(1)-N(3)	140.09(3)	C(2)-C(1)-C(6)	123.85(10)
O(3)-Gd(1)-N(3)	77.43(3)	C(3)-C(2)-C(1)	118.41(12)
N(1)-Gd(1)-N(3)	129.98(3)	C(3)-C(2)-H(2)	120.6(9)
N(5)-Gd(1)-N(3)	100.48(3)	C(1)-C(2)-H(2)	121.0(9)
O(7)-Gd(1)-N(2)	139.96(3)	C(4)-C(3)-C(2)	119.31(11)
O(5)-Gd(1)-N(2)	66.05(3)	C(4)-C(3)-H(3A)	117.3(10)
O(1)-Gd(1)-N(2)	74.30(3)	C(2)-C(3)-H(3A)	123.3(10)
O(3)-Gd(1)-N(2)	139.52(2)	C(3)-C(4)-C(5)	118.78(12)
N(1)-Gd(1)-N(2)	124.89(3)	C(3)-C(4)-H(4)	122.2(10)
N(5)-Gd(1)-N(2)	67.89(3)	C(5)-C(4)-H(4)	119.0(10)
N(3)-Gd(1)-N(2)	68.18(3)	N(1)-C(5)-C(4)	121.54(11)
O(7)-Gd(1)-N(4)	65.99(3)	N(1)-C(5)-C(7)	114.25(9)
O(5)-Gd(1)-N(4)	138.60(3)	C(4)-C(5)-C(7)	124.21(11)
O(1)-Gd(1)-N(4)	140.23(3)	O(4)-C(6)-O(3)	126.56(10)
O(3)-Gd(1)-N(4)	75.90(3)	O(4)-C(6)-C(1)	117.65(9)
N(1)-Gd(1)-N(4)	127.00(3)	O(3)-C(6)-C(1)	115.77(9)
N(5)-Gd(1)-N(4)	68.35(3)	O(2)-C(7)-O(1)	127.06(11)
N(3)-Gd(1)-N(4)	67.37(3)	O(2)-C(7)-C(5)	117.88(10)
N(2)-Gd(1)-N(4)	108.11(3)	O(1)-C(7)-C(5)	115.06(9)
C(7)-O(1)-Gd(1)	125.67(7)	N(2)-C(8)-C(9)	113.16(8)
C(6)-O(3)-Gd(1)	124.95(7)	N(2)-C(8)-H(8A)	108.6(9)
C(17)-O(5)-Gd(1)	122.58(6)	C(9)-C(8)-H(8A)	110.5(8)
C(19)-O(7)-Gd(1)	123.58(6)	N(2)-C(8)-H(8B)	108.0(8)
C(1)-N(1)-C(5)	120.03(9)	C(9)-C(8)-H(8B)	109.7(8)
C(1)-N(1)-Gd(1)	119.87(7)	H(8A)-C(8)-H(8B)	106.7(11)
C(5)-N(1)-Gd(1)	120.10(7)	N(3)-C(9)-C(8)	110.49(8)
C(16)-N(2)-C(8)	110.35(8)	N(3)-C(9)-H(9A)	111.5(8)
C(16)-N(2)-C(15)	110.09(8)	C(8)-C(9)-H(9A)	109.5(8)
C(8)-N(2)-C(15)	110.33(8)	N(3)-C(9)-H(9B)	105.7(8)
C(16)-N(2)-Gd(1)	106.12(6)	C(8)-C(9)-H(9B)	110.8(8)
C(8)-N(2)-Gd(1)	110.83(6)	H(9A)-C(9)-H(9B)	108.7(10)
C(15)-N(2)-Gd(1)	109.02(6)	N(3)-C(10)-C(11)	110.04(9)
C(10)-N(3)-C(9)	112.22(8)	N(3)-C(10)-H(10A)	111.7(8)
C(10)-N(3)-Gd(1)	115.73(6)	C(11)-C(10)-H(10A)	106.3(8)
C(9)-N(3)-Gd(1)	112.49(6)	N(3)-C(10)-H(10B)	109.1(8)
C(10)-N(3)-H(3)	105.8(9)	C(11)-C(10)-H(10B)	112.2(7)
C(9)-N(3)-H(3)	108.7(9)	H(10A)-C(10)-H(10B)	107.5(11)
Gd(1)-N(3)-H(3)	100.9(9)	N(4)-C(11)-C(10)	111.80(9)
C(18)-N(4)-C(12)	110.36(8)	N(4)-C(11)-H(11A)	110.3(7)
C(18)-N(4)-C(11)	110.15(9)	C(10)-C(11)-H(11A)	109.3(8)
C(12)-N(4)-C(11)	110.16(8)	N(4)-C(11)-H(11B)	105.6(7)
C(18)-N(4)-Gd(1)	106.16(6)	C(10)-C(11)-H(11B)	108.4(7)
C(12)-N(4)-Gd(1)	110.41(6)	H(11A)-C(11)-H(11B)	111.3(11)

N(4)-C(12)-C(13)	112.92(9)
N(4)-C(12)-H(12A)	111.1(8)
C(13)-C(12)-H(12A)	109.8(8)
N(4)-C(12)-H(12B)	106.8(7)
C(13)-C(12)-H(12B)	111.4(8)
H(12A)-C(12)-H(12B)	104.5(11)
N(5)-C(13)-C(12)	110.78(9)
N(5)-C(13)-H(13A)	111.4(9)
C(12)-C(13)-H(13A)	108.3(9)
N(5)-C(13)-H(13B)	108.3(8)
C(12)-C(13)-H(13B)	113.2(8)
H(13A)-C(13)-H(13B)	104.8(11)
N(5)-C(14)-C(15)	110.07(8)
N(5)-C(14)-H(14A)	111.7(8)
C(15)-C(14)-H(14A)	107.6(8)
N(5)-C(14)-H(14B)	109.4(8)
C(15)-C(14)-H(14B)	111.6(8)
H(14A)-C(14)-H(14B)	106.4(11)
N(2)-C(15)-C(14)	111.56(9)
N(2)-C(15)-H(15A)	111.1(7)
C(14)-C(15)-H(15A)	109.0(8)
N(2)-C(15)-H(15B)	106.4(8)
C(14)-C(15)-H(15B)	109.0(8)
H(15A)-C(15)-H(15B)	109.8(11)
N(2)-C(16)-C(17)	114.22(9)
N(2)-C(16)-H(16A)	111.7(9)
C(17)-C(16)-H(16A)	103.9(9)
N(2)-C(16)-H(16B)	110.4(8)
C(17)-C(16)-H(16B)	107.5(8)
H(16A)-C(16)-H(16B)	108.8(12)
O(6)-C(17)-O(5)	124.68(10)
O(6)-C(17)-C(16)	117.42(9)
O(5)-C(17)-C(16)	117.88(9)
N(4)-C(18)-C(19)	114.25(9)
N(4)-C(18)-H(18A)	107.5(8)
C(19)-C(18)-H(18A)	107.9(8)
N(4)-C(18)-H(18B)	109.8(9)
C(19)-C(18)-H(18B)	108.2(9)
H(18A)-C(18)-H(18B)	109.1(12)
O(8)-C(19)-O(7)	124.37(9)
O(8)-C(19)-C(18)	117.81(9)
O(7)-C(19)-C(18)	117.77(8)
C(29)-N(6)-C(21)	106.92(8)
C(29)-N(6)-C(25)	110.97(9)
C(21)-N(6)-C(25)	110.39(8)
C(29)-N(6)-C(33)	110.84(8)
C(21)-N(6)-C(33)	110.70(8)
C(25)-N(6)-C(33)	107.05(8)
C(22)-C(21)-N(6)	115.44(9)
C(21)-C(22)-C(23)	110.31(10)
C(22)-C(23)-C(24)	112.18(11)
N(6)-C(25)-C(26)	115.42(9)
C(27)-C(26)-C(25)	110.50(10)
C(26)-C(27)-C(28)	111.96(11)
N(6)-C(29)-C(30)	115.63(9)
C(31)-C(30)-C(29)	111.61(11)
C(30)-C(31)-C(32)	115.48(13)
C(34)-C(33)-N(6)	115.70(8)
C(33)-C(34)-C(35)	111.05(9)
C(36)-C(35)-C(34)	113.25(10)
H(21D)-O(21)-H(21C)	99.8(17)
H(22C)-O(22)-H(22D)	107.2(17)
O(51)-C(52)-C(53)	123.0(4)
O(51)-C(52)-C(51)	119.1(4)
C(53)-C(52)-C(51)	117.6(3)
O(61)-C(62)-C(64)	136.7(4)

Table 5. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for MLC13 (CCDC 746175). The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Gd(1)	125(1)	166(1)	111(1)	15(1)	12(1)	13(1)
O(1)	192(3)	237(3)	155(4)	33(3)	-3(3)	5(3)
O(2)	346(5)	310(4)	256(5)	142(3)	-71(4)	-35(3)
O(3)	194(3)	226(3)	136(4)	-7(3)	0(3)	23(3)
O(4)	708(7)	316(4)	158(4)	-72(3)	-88(4)	90(4)
O(5)	160(3)	269(4)	119(4)	-3(3)	20(3)	-11(3)
O(6)	189(3)	208(3)	156(4)	31(2)	36(3)	17(3)
O(7)	165(3)	243(3)	138(4)	29(3)	31(3)	25(3)
O(8)	278(4)	270(4)	164(4)	-11(3)	78(3)	15(3)
N(1)	130(3)	220(4)	154(4)	21(3)	34(3)	9(3)
N(2)	161(4)	212(4)	149(4)	4(3)	11(3)	-3(3)
N(3)	162(4)	220(4)	172(4)	9(3)	1(3)	23(3)
N(4)	181(4)	222(4)	174(4)	31(3)	11(3)	21(3)
N(5)	169(4)	203(4)	176(4)	13(3)	12(3)	7(3)
C(1)	147(4)	224(4)	184(5)	-8(3)	37(4)	14(3)
C(2)	276(6)	240(5)	313(7)	-54(4)	-25(5)	15(4)
C(3)	449(8)	216(5)	469(9)	-17(5)	-101(6)	22(5)
C(4)	342(6)	237(5)	408(8)	77(5)	-93(6)	10(5)
C(5)	161(4)	226(4)	233(6)	62(4)	11(4)	-4(3)
C(6)	213(5)	252(5)	135(5)	-20(3)	31(4)	28(4)
C(7)	163(4)	264(5)	187(5)	74(4)	4(4)	-16(4)
C(8)	197(5)	225(4)	209(5)	-41(4)	20(4)	37(4)
C(9)	172(5)	237(5)	211(6)	-14(4)	31(4)	49(4)
C(10)	223(5)	222(5)	248(6)	49(4)	14(4)	63(4)
C(11)	218(5)	250(5)	210(6)	76(4)	-4(4)	46(4)
C(12)	236(5)	222(5)	239(6)	59(4)	14(4)	-15(4)
C(13)	161(4)	233(5)	227(6)	23(4)	26(4)	-24(4)
C(14)	197(5)	222(4)	207(5)	-34(4)	6(4)	-32(4)
C(15)	185(5)	253(5)	168(5)	-33(4)	-5(4)	-10(4)
C(16)	194(5)	258(5)	135(5)	-5(3)	29(4)	-21(4)
C(17)	140(4)	199(4)	144(4)	4(3)	14(3)	40(3)
C(18)	215(5)	284(5)	141(5)	58(4)	42(4)	26(4)
C(19)	149(4)	206(4)	142(4)	1(3)	5(3)	-31(3)
N(6)	158(4)	254(4)	193(5)	98(3)	-12(3)	-19(3)
C(21)	191(5)	253(5)	203(5)	92(4)	-24(4)	-35(4)
C(22)	314(6)	342(6)	213(6)	61(5)	-39(5)	-66(5)
C(23)	298(6)	324(6)	262(6)	31(4)	2(5)	-61(5)
C(24)	447(8)	429(7)	333(8)	-29(6)	-16(6)	-93(6)
C(25)	174(5)	312(5)	269(6)	165(4)	-27(4)	-10(4)
C(26)	176(5)	405(7)	325(7)	198(5)	-7(5)	-2(5)
C(27)	193(6)	742(11)	809(14)	569(10)	-6(7)	53(6)
C(28)	191(6)	761(12)	825(15)	432(10)	6(7)	76(7)
C(29)	209(5)	241(5)	197(5)	69(4)	-7(4)	2(4)
C(30)	397(7)	290(6)	344(8)	50(5)	-16(6)	-92(5)
C(31)	560(9)	289(6)	379(9)	-10(5)	-107(7)	51(6)

C(32)	705(12)	419(9)	708(14)	-159(8)	-102(10)	-110(8)
C(33)	184(5)	301(5)	195(5)	99(4)	2(4)	-38(4)
C(34)	194(5)	391(6)	219(6)	115(4)	19(4)	18(4)
C(35)	262(6)	324(6)	248(6)	67(4)	57(5)	7(4)
C(36)	250(6)	419(7)	434(9)	83(6)	98(6)	53(5)
O(21)	377(5)	260(4)	174(4)	-7(3)	66(3)	70(3)
O(22)	446(6)	442(5)	223(5)	46(4)	66(4)	-175(4)
O(51)	1028(17)	250(8)	839(18)	80(9)	-287(14)	41(9)
C(51)	427(17)	2080(50)	580(20)	650(30)	-221(15)	-510(20)
C(52)	1170(30)	184(9)	740(20)	-39(11)	-640(20)	169(13)
C(53)	216(10)	577(15)	556(19)	-171(12)	-51(10)	59(9)
O(61)	565(17)	560(16)	236(14)	-88(11)	-133(12)	369(13)
C(62)	580(30)	600(30)	1060(50)	-340(30)	240(30)	-270(20)
C(64)	570(30)	650(30)	950(50)	-480(30)	-140(30)	40(20)
O(71)	253(11)	270(10)	238(12)	-85(8)	60(8)	-11(8)

Table 6. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for MLC13 (CCDC 746175) (refined hydrogens only).

	x	y	z	U_{iso}
H(3)	9397(11)	3996(11)	5332(6)	31(4)
H(5)	5627(10)	4286(11)	4588(5)	26(4)
H(2)	8179(10)	8438(11)	5965(6)	32(4)
H(3A)	7548(11)	9482(12)	5295(6)	35(4)
H(4)	6863(11)	8790(12)	4562(6)	41(4)
H(8A)	8947(10)	2974(10)	3930(6)	25(3)
H(8B)	8220(10)	2567(10)	4330(5)	24(3)
H(9A)	9866(9)	2771(9)	4683(5)	19(3)
H(9B)	9841(9)	3915(10)	4551(5)	17(3)
H(10A)	9260(10)	2436(11)	5592(5)	27(4)
H(10B)	8347(9)	2240(10)	5216(5)	19(3)
H(11A)	7818(9)	2467(10)	6078(5)	19(3)
H(11B)	8321(8)	3526(9)	6078(5)	9(3)
H(12A)	5975(10)	2392(10)	5742(5)	25(3)
H(12B)	6756(9)	2282(10)	5288(5)	22(3)
H(13A)	5131(10)	2734(12)	4987(5)	31(4)
H(13B)	5073(10)	3701(10)	5282(5)	23(3)
H(14A)	5706(10)	2898(10)	4069(5)	23(3)
H(14B)	6614(9)	2525(10)	4386(5)	21(3)
H(15A)	7143(10)	3199(10)	3585(5)	25(3)
H(15B)	6665(9)	4219(9)	3761(5)	16(3)
H(16A)	7997(11)	5136(10)	3627(6)	27(4)
H(16B)	8917(10)	4429(10)	3564(5)	21(3)
H(18A)	7078(11)	4306(11)	6362(6)	35(4)
H(18B)	6086(11)	3621(11)	6301(6)	34(4)
H(21D)	410(13)	5370(14)	3322(7)	45(5)
H(21C)	956(12)	4616(14)	3264(7)	42(5)
H(22C)	4487(12)	4446(13)	6737(6)	36(4)
H(22D)	3998(13)	3590(15)	6653(7)	54(5)

Table 7. Hydrogen bonds for MLC13 (CCDC 746175) [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(3)-H(3)...O(5)#1	0.956(15)	2.249(14)	3.0722(12)	143.7(12)
N(3)-H(3)...O(6)#1	0.956(15)	2.442(15)	3.2454(13)	141.5(12)
N(5)-H(5)...O(7)#2	0.911(14)	2.286(14)	3.0535(12)	141.7(11)
N(5)-H(5)...O(8)#2	0.911(14)	2.614(14)	3.2907(13)	131.6(11)
O(21)-H(21D)...O(6)#3	0.846(18)	1.952(19)	2.7966(11)	177.2(17)
O(21)-H(21C)...O(4)#2	0.793(18)	2.089(19)	2.8783(13)	173.6(17)
O(22)-H(22C)...O(8)	0.825(17)	2.042(17)	2.8533(12)	167.8(16)
O(22)-H(22D)...O(2)#2	0.836(19)	2.04(2)	2.8755(15)	177.1(19)
O(61)-H(61)...O(71)	0.84	2.36	2.841(3)	116.7

Symmetry transformations used to generate equivalent atoms:
#1 -x+2,-y+1,-z+1
#2 -x+1,-y+1,-z+1
#3 x-1,y,z

CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Date 4 December 2004

Crystal Structure Analysis of:

MLC03

(shown below)

For Investigator: Morgan Cable ext. (818) 354-4348

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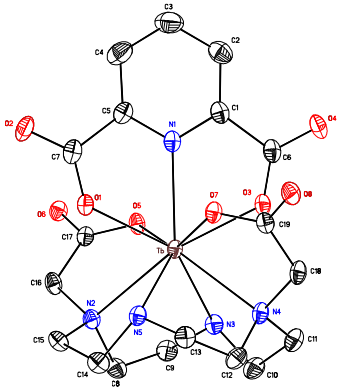
Contents



MLC03

Note: The crystallographic data have been deposited in the Cambridge Database (CCDC) and has been placed on hold pending further instructions from me. The deposition number is 629534. Ideally the CCDC would like the publication to contain a footnote of the type: "Crystallographic data have been deposited at the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK and copies can be obtained on request, free of charge, by quoting the publication citation and the deposition number 629354."

Table 1. Crystal data and structure refinement for MLC03 (CCDC 629534).

Empirical formula	$[C_{19}H_{25}N_5O_8Tb]^-[C_{16}H_{36}N]^+ \bullet 0.47(C_3H_8O)$
$0.53(C_3H_6O)\ 3(H_2O)$	
Formula weight	964.94
Crystallization Solvent	Acetone/isopropanol/water
Crystal Habit	Square
Crystal size	0.35 x 0.31 x 0.11 mm^3
Crystal color	Colorless

Data Collection

Type of diffractometer	Bruker SMART 1000
Wavelength	0.71073 Å MoKα
Data Collection Temperature	100(2) K
θ range for 24638 reflections used in lattice determination	2.18 to 37.67°
Unit cell dimensions	a = 13.1047(5) Å b = 13.3397(5) Å β= 90.0130(10)° c = 26.0901(9) Å
Volume	4560.9(3) $Å^3$
Z	4
Crystal system	Monoclinic
Space group	$P2_1/c$
Density (calculated)	1.405 Mg/m^3
F(000)	2016
θ range for data collection	1.56 to 37.67°
Completeness to θ = 37.67°	83.6 %
Index ranges	$-22 \le h \le 22, -17 \le k \le 20, -40 \le l \le 44$
Data collection scan type	ω scans at 5 ϕ settings
Reflections collected	78733
Independent reflections	20316 [R_{int}= 0.0879]
Absorption coefficient	1.613 mm^{-1}
Absorption correction	None
Max. and min. transmission	0.8425 and 0.6022

Table 1 (cont.)

Structure solution and Refinement

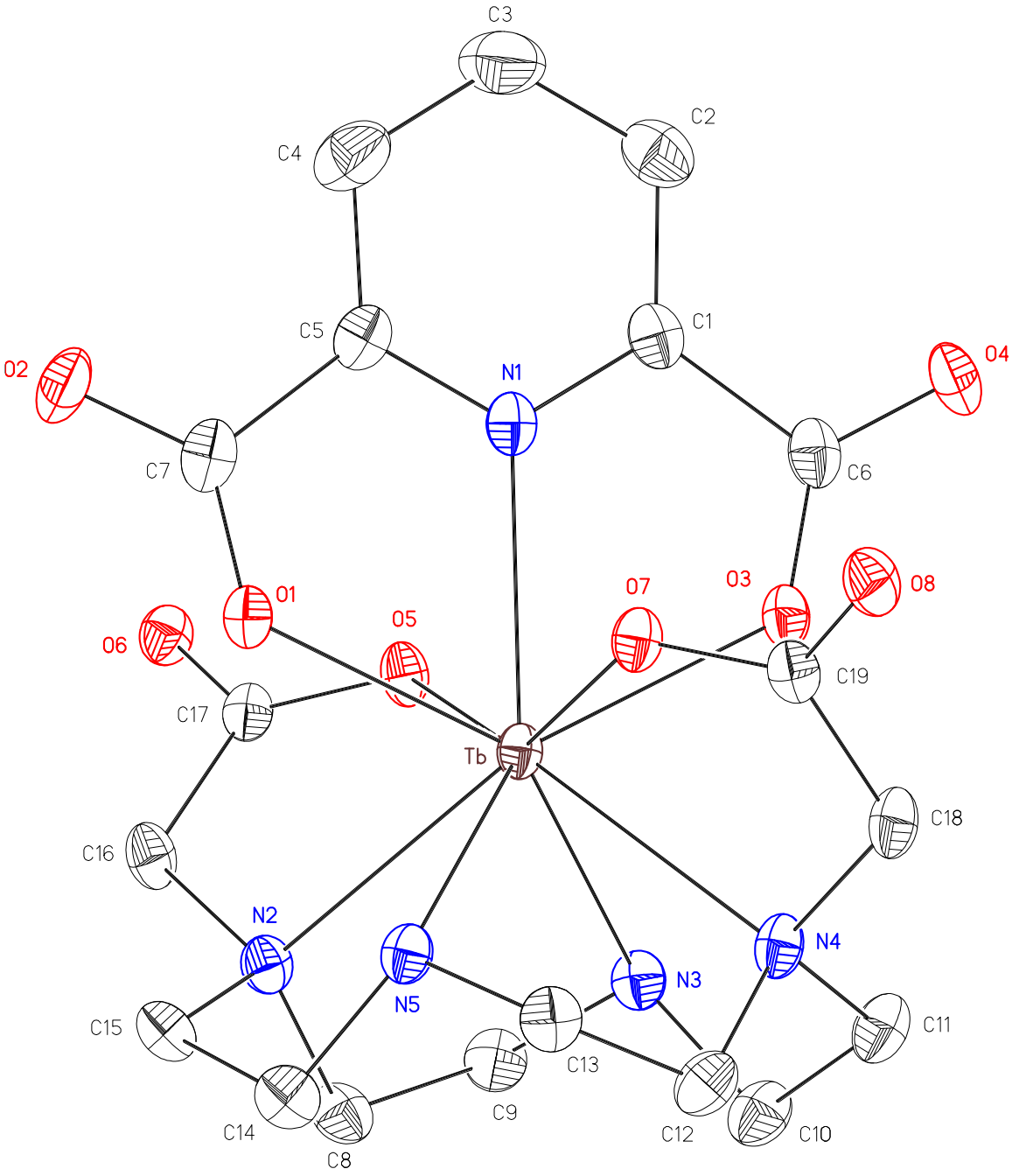
Structure solution program	Bruker XS v6.12
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	Bruker XL v6.12
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	20316 / 0 / 577
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	1.077
Final R indices [$I>2\sigma(I)$, 11252 reflections]	R1 = 0.0384, *w*R2 = 0.0639
R indices (all data)	R1 = 0.0900, *w*R2 = 0.0717
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.005
Average shift/error	0.000
Largest diff. peak and hole	3.035 and -2.004 e.Å^{-3}

Special Refinement Details

The crystals contain a disordered solvent. The site contains acetone and water or isopropanol and water, modeled as a combination of both with a total occupancy = 1.0.

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.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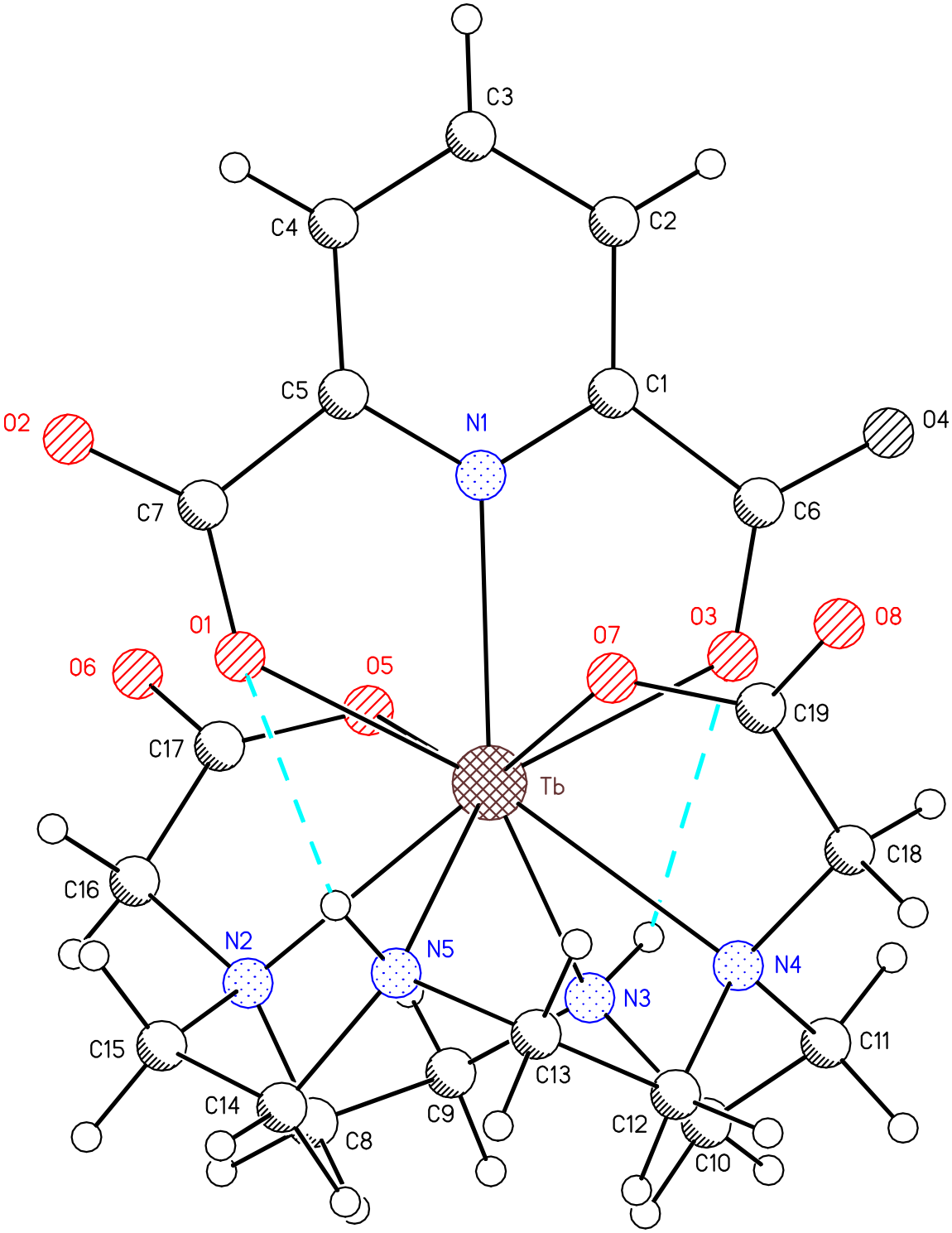
C2
C4
C5
C1
O2
N1
O4
C7
C6
O1
O7
O3
O8
O6
O5
C19
C17
Tb
C18
C16
N2
N4
N5
N3
C15
C11
C13
C9
C14
C12
C10
C8

D55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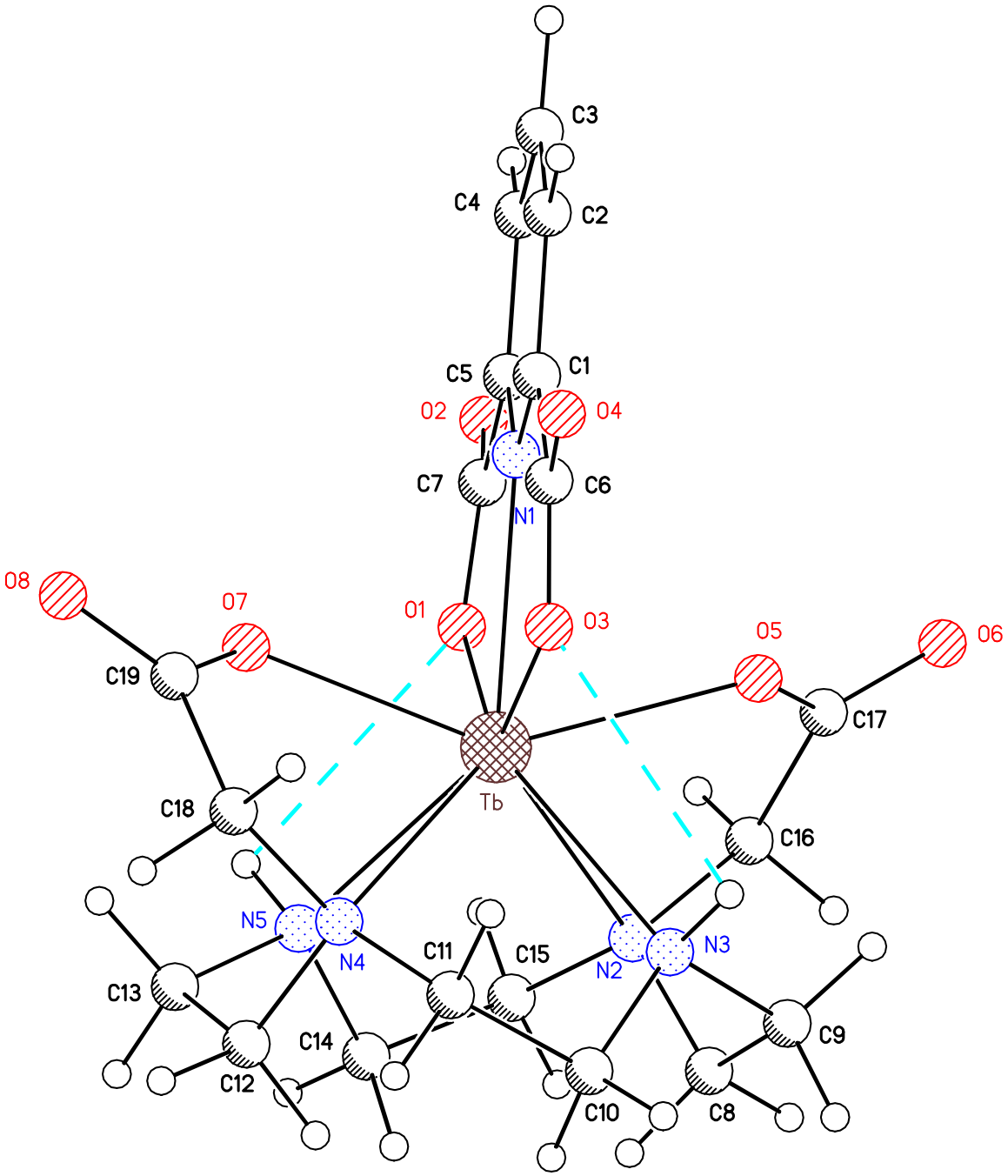
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C19
C17
Tb
C18
C16
N5
N4
C11
C15
N2
N3
C13
C9
C14
C12
C10
C8

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for MLC03 (CCDC 629534). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}	Occ
Tb	2488(1)	77(1)	4995(1)	14(1)	1
O(1)	1702(1)	-870(1)	5664(1)	19(1)	1
O(2)	1420(1)	-2322(1)	6074(1)	29(1)	1
O(3)	3304(1)	-500(1)	4227(1)	19(1)	1
O(4)	3642(2)	-1709(1)	3658(1)	36(1)	1
O(5)	3947(1)	-573(1)	5407(1)	18(1)	1
O(6)	4672(1)	-1065(1)	6137(1)	19(1)	1
O(7)	1058(1)	-336(1)	4497(1)	18(1)	1
O(8)	303(1)	-391(1)	3733(1)	24(1)	1
N(1)	2504(1)	-1777(1)	4876(1)	16(1)	1
N(2)	2951(1)	1011(1)	5858(1)	17(1)	1
N(3)	3914(1)	1368(1)	4861(1)	18(1)	1
N(4)	2027(1)	1446(2)	4294(1)	18(1)	1
N(5)	1048(1)	1222(1)	5302(1)	18(1)	1
C(1)	2893(2)	-2167(2)	4449(1)	19(1)	1
C(2)	2910(2)	-3195(2)	4365(1)	28(1)	1
C(3)	2534(2)	-3821(2)	4746(1)	38(1)	1
C(4)	2134(2)	-3404(2)	5191(1)	33(1)	1
C(5)	2135(2)	-2372(2)	5242(1)	20(1)	1
C(6)	3313(2)	-1395(2)	4073(1)	21(1)	1
C(7)	1713(2)	-1817(2)	5707(1)	21(1)	1
C(8)	3637(2)	1866(2)	5759(1)	21(1)	1
C(9)	4425(2)	1648(2)	5351(1)	22(1)	1
C(10)	3663(2)	2248(2)	4549(1)	24(1)	1
C(11)	2970(2)	1931(2)	4108(1)	23(1)	1
C(12)	1324(2)	2205(2)	4513(1)	24(1)	1
C(13)	542(2)	1760(2)	4874(1)	21(1)	1
C(14)	1301(2)	1900(2)	5731(1)	22(1)	1
C(15)	1991(2)	1367(2)	6106(1)	20(1)	1
C(16)	3455(2)	255(2)	6184(1)	20(1)	1
C(17)	4081(2)	-512(2)	5889(1)	16(1)	1
C(18)	1525(2)	894(2)	3870(1)	22(1)	1
C(19)	904(2)	-2(2)	4045(1)	18(1)	1
N(6)	6377(1)	2839(1)	2800(1)	20(1)	1
C(20)	7192(2)	3471(2)	2542(1)	24(1)	1
C(21)	8271(2)	3325(2)	2746(1)	29(1)	1
C(22)	8999(2)	4044(3)	2481(1)	55(1)	1
C(23)	10110(2)	3815(3)	2629(1)	57(1)	1
C(24)	6673(2)	1733(2)	2794(1)	22(1)	1
C(25)	6831(2)	1282(2)	2265(1)	30(1)	1
C(26)	7071(2)	176(2)	2308(1)	31(1)	1
C(27)	7281(2)	-300(2)	1788(1)	39(1)	1
C(28)	5383(2)	3004(2)	2504(1)	24(1)	1
C(29)	4458(2)	2451(2)	2707(1)	27(1)	1
C(30)	3606(2)	2431(2)	2311(1)	28(1)	1
C(31)	2648(2)	1905(2)	2501(1)	37(1)	1

C(32)	6243(2)	3133(2)	3359(1)	21(1)	1
C(33)	5990(2)	4229(2)	3451(1)	32(1)	1
C(34)	5677(2)	4402(2)	3999(1)	37(1)	1
C(35)	5560(3)	5486(3)	4151(1)	58(1)	1
O(11)	5754(1)	5055(1)	1892(1)	27(1)	1
O(12)	9175(2)	975(2)	3123(1)	35(1)	1
O(51A)	1484(7)	4416(5)	6143(5)	65(3)	0.476(4)
C(52A)	544(6)	4415(7)	6202(4)	64(3)	0.476(4)
C(53A)	2(6)	3743(6)	6551(3)	58(2)	0.476(4)
C(54A)	-223(9)	4727(10)	5781(5)	92(4)	0.476(4)
O(50A)	8868(4)	2675(4)	1469(2)	62(2)	0.476(4)
O(51B)	1315(5)	4316(3)	5364(2)	75(2)	0.524(4)
C(52B)	899(7)	4533(5)	5758(3)	53(2)	0.524(4)
C(53B)	-22(8)	5292(7)	5909(5)	68(3)	0.524(4)
C(54B)	1385(15)	4504(15)	6255(7)	149(10)	0.524(4)
O(50B)	-123(5)	3726(6)	7096(2)	96(2)	0.524(4)

Table 3. Bond lengths [Å] and angles [°] for MLC03 (CCDC 629534).

Tb-O(7)	2.3467(14)	C(26)-C(27)	1.522(4)
Tb-O(5)	2.3573(15)	C(28)-C(29)	1.515(3)
Tb-O(1)	2.3879(15)	C(29)-C(30)	1.522(3)
Tb-O(3)	2.3987(15)	C(30)-C(31)	1.522(4)
Tb-N(1)	2.4918(19)	C(32)-C(33)	1.517(3)
Tb-N(5)	2.5564(18)	C(33)-C(34)	1.505(4)
Tb-N(3)	2.5657(18)	C(34)-C(35)	1.507(4)
Tb-N(2)	2.6437(17)	O(51A)-C(52A)	1.241(12)
Tb-N(4)	2.6550(18)	C(52A)-C(53A)	1.463(12)
O(1)-C(7)	1.267(3)	C(52A)-C(54A)	1.545(15)
O(2)-C(7)	1.234(3)	O(51B)-C(52B)	1.199(8)
O(3)-C(6)	1.259(3)	C(52B)-C(54B)	1.445(19)
O(4)-C(6)	1.241(3)	C(52B)-C(53B)	1.625(14)
O(5)-C(17)	1.272(2)		
O(6)-C(17)	1.250(2)	O(7)-Tb-O(5)	144.47(6)
O(7)-C(19)	1.276(2)	O(7)-Tb-O(1)	86.35(5)
O(8)-C(19)	1.245(2)	O(5)-Tb-O(1)	79.79(5)
N(1)-C(5)	1.332(3)	O(7)-Tb-O(3)	79.49(5)
N(1)-C(1)	1.332(3)	O(5)-Tb-O(3)	84.35(5)
N(2)-C(8)	1.475(3)	O(1)-Tb-O(3)	129.31(6)
N(2)-C(16)	1.475(3)	O(7)-Tb-N(1)	72.84(5)
N(2)-C(15)	1.491(3)	O(5)-Tb-N(1)	71.66(5)
N(3)-C(10)	1.466(3)	O(1)-Tb-N(1)	64.54(5)
N(3)-C(9)	1.489(3)	O(3)-Tb-N(1)	64.78(6)
N(4)-C(18)	1.483(3)	O(7)-Tb-N(5)	74.00(5)
N(4)-C(12)	1.482(3)	O(5)-Tb-N(5)	132.49(5)
N(4)-C(11)	1.478(3)	O(1)-Tb-N(5)	76.65(6)
N(5)-C(13)	1.482(3)	O(3)-Tb-N(5)	141.46(5)
N(5)-C(14)	1.475(3)	N(1)-Tb-N(5)	129.68(6)
C(1)-C(2)	1.389(3)	O(7)-Tb-N(3)	131.59(5)
C(1)-C(6)	1.525(3)	O(5)-Tb-N(3)	73.61(6)
C(2)-C(3)	1.390(4)	O(1)-Tb-N(3)	140.27(5)
C(3)-C(4)	1.389(4)	O(3)-Tb-N(3)	77.08(6)
C(4)-C(5)	1.384(3)	N(1)-Tb-N(3)	129.99(6)
C(5)-C(7)	1.525(3)	N(5)-Tb-N(3)	100.32(6)
C(8)-C(9)	1.513(3)	O(7)-Tb-N(2)	139.96(5)
C(10)-C(11)	1.526(3)	O(5)-Tb-N(2)	66.38(5)
C(12)-C(13)	1.515(3)	O(1)-Tb-N(2)	74.10(5)
C(14)-C(15)	1.510(3)	O(3)-Tb-N(2)	139.53(5)
C(16)-C(17)	1.522(3)	N(1)-Tb-N(2)	124.89(5)
C(18)-C(19)	1.517(3)	N(5)-Tb-N(2)	67.71(5)
N(6)-C(20)	1.518(3)	N(3)-Tb-N(2)	68.45(6)
N(6)-C(28)	1.528(3)	O(7)-Tb-N(4)	66.30(5)
N(6)-C(32)	1.522(3)	O(5)-Tb-N(4)	138.69(5)
N(6)-C(24)	1.526(3)	O(1)-Tb-N(4)	140.29(5)
C(20)-C(21)	1.524(3)	O(3)-Tb-N(4)	75.30(6)
C(21)-C(22)	1.518(4)	N(1)-Tb-N(4)	126.78(5)
C(22)-C(23)	1.538(4)	N(5)-Tb-N(4)	68.67(6)
C(24)-C(25)	1.519(3)	N(3)-Tb-N(4)	67.04(5)
C(25)-C(26)	1.513(4)	N(2)-Tb-N(4)	108.32(6)

C(7)-O(1)-Tb	125.87(15)	N(4)-C(18)-C(19)	113.87(17)
C(6)-O(3)-Tb	125.03(14)	O(8)-C(19)-O(7)	123.9(2)
C(17)-O(5)-Tb	122.63(14)	O(8)-C(19)-C(18)	118.11(19)
C(19)-O(7)-Tb	123.76(14)	O(7)-C(19)-C(18)	117.97(18)
C(5)-N(1)-C(1)	120.4(2)	C(20)-N(6)-C(28)	107.20(16)
C(5)-N(1)-Tb	119.92(14)	C(20)-N(6)-C(32)	111.30(18)
C(1)-N(1)-Tb	119.72(15)	C(28)-N(6)-C(32)	110.36(17)
C(8)-N(2)-C(16)	110.85(17)	C(20)-N(6)-C(24)	110.72(17)
C(8)-N(2)-C(15)	110.12(18)	C(28)-N(6)-C(24)	110.53(18)
C(16)-N(2)-C(15)	110.26(17)	C(32)-N(6)-C(24)	106.77(16)
C(8)-N(2)-Tb	110.80(13)	N(6)-C(20)-C(21)	115.28(18)
C(16)-N(2)-Tb	105.73(13)	C(20)-C(21)-C(22)	110.1(2)
C(15)-N(2)-Tb	108.99(12)	C(23)-C(22)-C(21)	110.8(2)
C(10)-N(3)-C(9)	112.04(18)	C(25)-C(24)-N(6)	115.25(18)
C(10)-N(3)-Tb	116.74(13)	C(26)-C(25)-C(24)	110.4(2)
C(9)-N(3)-Tb	112.29(13)	C(25)-C(26)-C(27)	112.2(2)
C(18)-N(4)-C(12)	110.54(17)	N(6)-C(28)-C(29)	115.84(18)
C(18)-N(4)-C(11)	110.08(17)	C(30)-C(29)-C(28)	110.94(19)
C(12)-N(4)-C(11)	110.32(19)	C(29)-C(30)-C(31)	113.0(2)
C(18)-N(4)-Tb	105.86(13)	C(33)-C(32)-N(6)	115.22(18)
C(12)-N(4)-Tb	110.24(12)	C(34)-C(33)-C(32)	111.0(2)
C(11)-N(4)-Tb	109.71(12)	C(33)-C(34)-C(35)	115.1(3)
C(13)-N(5)-C(14)	111.97(18)	O(51A)-C(52A)-C(53A)	124.1(10)
C(13)-N(5)-Tb	112.57(13)	O(51A)-C(52A)-C(54A)	123.9(11)
C(14)-N(5)-Tb	115.99(13)	C(53A)-C(52A)-C(54A)	106.9(8)
N(1)-C(1)-C(2)	121.7(2)	O(51B)-C(52B)-C(54B)	124.3(12)
N(1)-C(1)-C(6)	114.3(2)	O(51B)-C(52B)-C(53B)	134.0(8)
C(2)-C(1)-C(6)	124.0(2)	C(54B)-C(52B)-C(53B)	97.3(10)
C(1)-C(2)-C(3)	118.2(2)		
C(2)-C(3)-C(4)	119.5(2)		
C(5)-C(4)-C(3)	118.5(2)		
N(1)-C(5)-C(4)	121.7(2)		
N(1)-C(5)-C(7)	114.3(2)		
C(4)-C(5)-C(7)	124.0(2)		
O(4)-C(6)-O(3)	127.0(2)		
O(4)-C(6)-C(1)	117.3(2)		
O(3)-C(6)-C(1)	115.66(19)		
O(2)-C(7)-O(1)	127.5(2)		
O(2)-C(7)-C(5)	117.8(2)		
O(1)-C(7)-C(5)	114.7(2)		
N(2)-C(8)-C(9)	113.07(19)		
N(3)-C(9)-C(8)	110.25(17)		
N(3)-C(10)-C(11)	109.31(19)		
N(4)-C(11)-C(10)	111.82(18)		
N(4)-C(12)-C(13)	113.11(19)		
N(5)-C(13)-C(12)	110.81(17)		
N(5)-C(14)-C(15)	109.68(18)		
N(2)-C(15)-C(14)	112.02(18)		
N(2)-C(16)-C(17)	114.21(17)		
O(6)-C(17)-O(5)	124.0(2)		
O(6)-C(17)-C(16)	118.02(18)		
O(5)-C(17)-C(16)	117.91(18)		

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for MLC03 (CCDC 629534). The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Tb	118(1)	186(1)	123(1)	15(1)	-14(1)	-8(1)
O(1)	184(8)	233(9)	143(7)	17(6)	1(6)	7(7)
O(2)	338(10)	303(10)	234(9)	121(7)	73(7)	31(8)
O(3)	182(8)	248(9)	127(7)	6(6)	-12(6)	-19(6)
O(4)	580(13)	351(11)	155(8)	-71(8)	75(8)	-83(9)
O(5)	157(7)	258(9)	123(7)	-2(6)	-28(5)	12(6)
O(6)	174(8)	216(8)	168(7)	18(6)	-30(6)	-12(6)
O(7)	142(7)	244(8)	152(7)	29(6)	-22(5)	-2(6)
O(8)	246(9)	292(9)	166(7)	-15(7)	-65(6)	4(7)
N(1)	112(8)	239(9)	143(8)	14(6)	-35(7)	4(7)
N(2)	162(9)	207(10)	150(8)	0(7)	-30(7)	10(7)
N(3)	137(9)	215(10)	179(9)	2(8)	9(7)	-12(7)
N(4)	148(9)	247(10)	150(8)	37(7)	-10(7)	-20(7)
N(5)	141(9)	213(10)	170(9)	22(8)	6(7)	-13(7)
C(1)	136(10)	270(12)	171(10)	-17(9)	-36(8)	-6(9)
C(2)	243(12)	300(14)	303(13)	-69(11)	25(10)	2(10)
C(3)	416(17)	250(15)	478(17)	-55(13)	105(13)	-9(12)
C(4)	309(14)	267(14)	414(16)	83(12)	92(12)	10(11)
C(5)	151(10)	241(12)	216(11)	49(9)	-11(8)	12(9)
C(6)	152(10)	308(13)	155(10)	-2(10)	-30(8)	-32(9)
C(7)	113(10)	306(13)	201(11)	59(10)	-9(8)	32(9)
C(8)	181(11)	245(12)	216(11)	-46(9)	-26(8)	-44(9)
C(9)	167(11)	252(12)	226(11)	-6(10)	-36(9)	-66(9)
C(10)	201(11)	251(13)	259(12)	46(10)	-18(9)	-65(9)
C(11)	202(11)	276(13)	224(11)	72(10)	-2(9)	-38(9)
C(12)	220(12)	231(12)	256(12)	70(10)	-33(9)	32(9)
C(13)	160(11)	256(12)	226(11)	10(10)	-25(9)	27(9)
C(14)	160(11)	245(12)	254(12)	-14(10)	-6(9)	42(9)
C(15)	185(11)	235(12)	193(10)	-54(9)	-11(8)	11(9)
C(16)	182(10)	278(12)	133(9)	-11(8)	-38(7)	34(9)
C(17)	120(9)	208(11)	153(9)	13(9)	-13(7)	-45(8)
C(18)	204(11)	303(13)	146(10)	48(9)	-29(8)	-12(9)
C(19)	142(9)	238(12)	164(8)	-3(9)	-1(6)	54(9)
N(6)	151(9)	263(10)	176(9)	86(8)	15(7)	23(7)
C(20)	154(11)	300(13)	256(12)	139(10)	31(8)	18(9)
C(21)	173(12)	375(16)	332(13)	199(12)	14(9)	8(11)
C(22)	179(13)	740(20)	720(20)	480(19)	2(13)	-43(14)
C(23)	218(15)	720(20)	780(30)	370(20)	17(15)	-79(15)
C(24)	182(11)	271(12)	193(10)	86(9)	15(8)	44(9)
C(25)	316(14)	352(16)	221(12)	65(11)	2(10)	79(12)
C(26)	306(13)	332(15)	279(12)	26(11)	-14(9)	50(11)
C(27)	392(16)	448(17)	327(14)	-37(13)	0(11)	76(13)
C(28)	178(11)	324(14)	203(11)	101(10)	-22(8)	37(9)
C(29)	185(11)	420(15)	196(11)	95(10)	-20(8)	-9(10)
C(30)	261(13)	329(14)	255(12)	56(10)	-65(10)	-30(10)
C(31)	257(14)	410(17)	454(17)	71(13)	-97(12)	-50(12)

C(32)	183(11)	257(12)	197(10)	66(9)	6(8)	-12(9)
C(33)	340(14)	316(14)	303(13)	73(11)	26(11)	57(11)
C(34)	448(17)	310(16)	353(15)	-16(12)	75(12)	-32(13)
C(35)	660(20)	440(20)	630(20)	-129(18)	116(19)	70(18)
O(11)	356(9)	269(9)	183(7)	10(7)	-68(6)	64(8)
O(12)	394(11)	454(12)	199(8)	34(8)	-58(7)	154(9)
O(51A)	280(30)	400(30)	1260(100)	-200(40)	160(40)	-40(20)
C(52A)	350(40)	770(60)	800(60)	-360(50)	70(40)	60(40)
C(53A)	480(40)	610(50)	640(50)	-180(40)	20(40)	90(40)
C(54A)	510(70)	1300(130)	960(100)	200(90)	90(60)	60(80)
O(50A)	620(30)	720(40)	520(30)	140(30)	-120(20)	40(30)
O(51B)	1160(50)	330(30)	740(40)	70(20)	380(30)	-30(30)
C(52B)	800(60)	230(30)	550(40)	40(30)	210(40)	-90(30)
C(53B)	380(50)	750(70)	900(70)	180(60)	-60(40)	-20(40)
C(54B)	990(140)	2900(200)	550(60)	-610(100)	-160(70)	390(130)
O(50B)	1110(50)	1170(60)	580(40)	-170(40)	230(30)	-350(40)

Table 5. Hydrogen bonds for MLC03 (CCDC 629534) [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(3)-H(3)...O(5)#1	0.93	2.25	3.078(2)	148.5
N(3)-H(3)...O(6)#1	0.93	2.43	3.221(2)	142.2
N(5)-H(5)...O(7)#2	0.93	2.20	3.048(2)	150.4
N(5)-H(5)...O(8)#2	0.93	2.51	3.271(3)	139.4
O(11)-H(11A)...O(4)#3	1.00	1.87	2.867(2)	172.1
O(11)-H(11B)...O(6)#4	1.00	1.82	2.776(2)	159.2
O(12)-H(12A)...O(2)#1	1.00	1.88	2.868(3)	170.6
O(12)-H(12B)...O(8)#5	1.00	1.88	2.836(2)	159.2
Other possible H-O interactions:				
N(3)-H(3)...O(3)	0.93	2.76	3.096(2)	102.2
N(5)-H(5)...O(1)	0.93	2.75	3.069(2)	101.3

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

CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Date 11 April 2007

Crystal Structure Analysis of:

MLC06

(shown below)

For Investigator: Morgan Cable ext. (818) 354-4345

Advisor: A. Ponce/H. B. Gray ext. 6500

Account Number: API.HSARPA-1-DOI.000002

By Michael W. Day 116 Beckman ext. 2734

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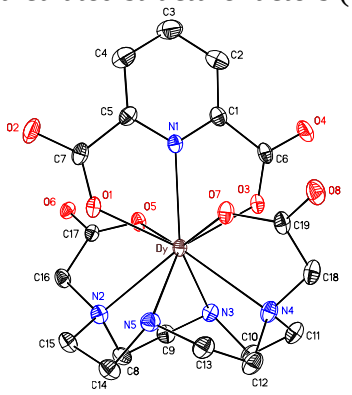
Contents



MLC06

Note: The crystallographic data have been deposited in the Cambridge Database (CCDC) and has been placed on hold pending further instructions from me. The deposition number is 643596. Ideally the CCDC would like the publication to contain a footnote of the type: "Crystallographic data have been deposited at the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK and copies can be obtained on request, free of charge, by quoting the publication citation and the deposition number 643596."

Table 1. Crystal data and structure refinement for MLC06 (CCDC 643596).

Empirical formula	$[C_{19}H_{25}N_5O_8Dy]^-$ $[C_{16}H_{36}N]^+$ $2(C_3H_8O)$ $3(H_2O)$
Formula weight	966.51
Crystallization Solvent	Acetone/isopropanol/water
Crystal Habit	Prism
Crystal size	0.39 x 0.14 x 0.11 mm^3
Crystal color	Colorless

Data Collection

Type of diffractometer	Bruker SMART 1000
Wavelength	0.71073 Å MoKα
Data Collection Temperature	100(2) K
θ range for 28613 reflections used in lattice determination	2.19 to 40.17°
Unit cell dimensions	a = 13.1742(4) Å b = 13.1860(4) Å β= 90.3720(10)° c = 26.1130(8) Å
Volume	4536.1(2) $Å^3$
Z	4
Crystal system	Monoclinic
Space group	$P2_1/c$
Density (calculated)	1.415 Mg/m^3
F(000)	2012
θ range for data collection	1.55 to 40.83°
Completeness to θ = 40.83°	93.8 %
Index ranges	$-23 \le h \le 23$, $-23 \le k \le 23$, $-46 \le l \le 48$
Data collection scan type	ω scans at 7 ϕ settings
Reflections collected	139731
Independent reflections	27766 [R_{int}= 0.1106]
Absorption coefficient	1.710 mm^{-1}
Absorption correction	None
Max. and min. transmission	0.8342 and 0.5553

Table 1 (cont.)

Structure solution and Refinement

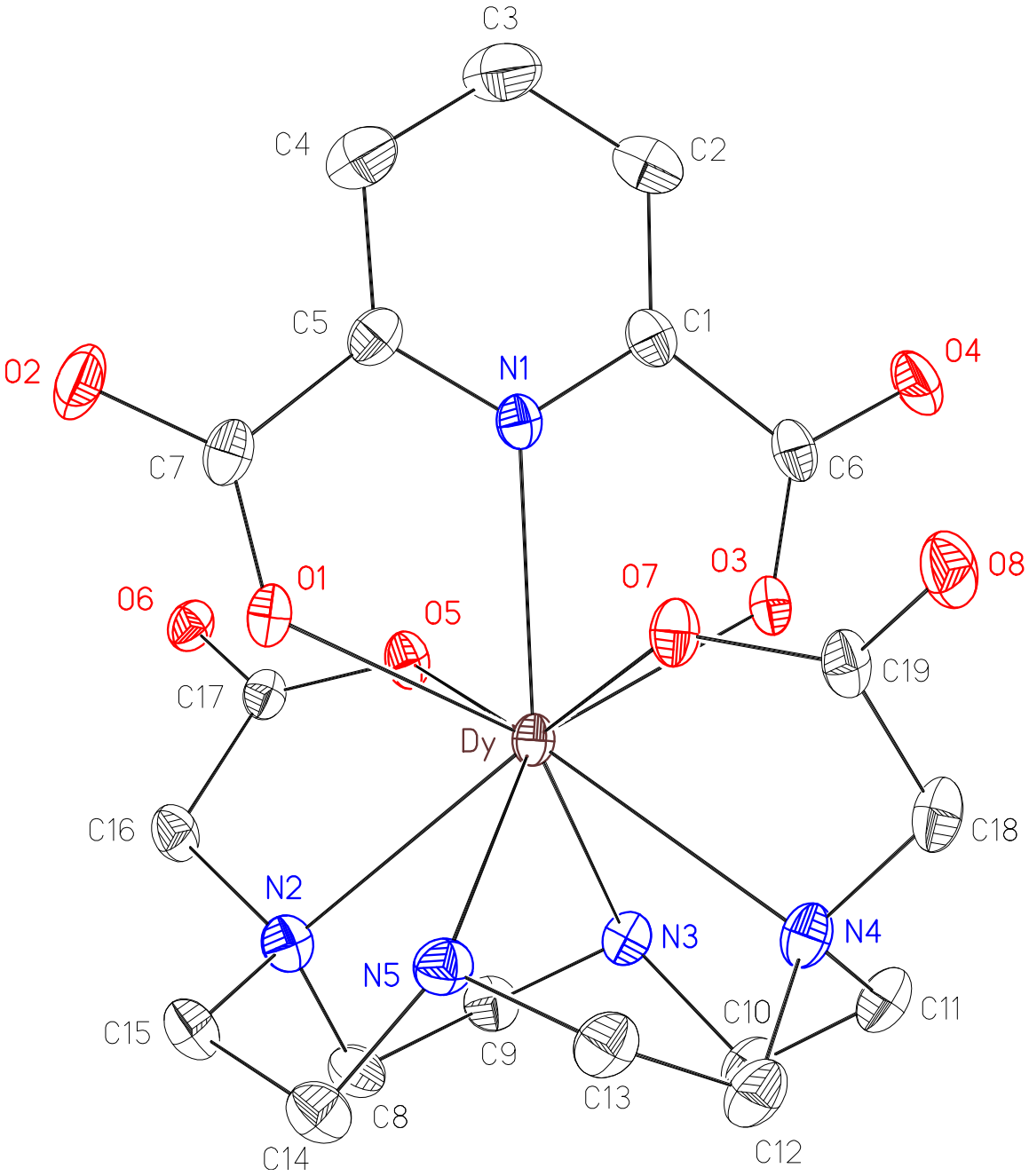
Structure solution program	SHELXS-97 (Sheldrick, 1990)
Primary solution method	Isomorphous method
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	SHELXL-97 (Sheldrick, 1997)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	27766 / 10 / 548
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	1.029
Final R indices [$I>2\sigma(I)$, 14774 reflections]	R1 = 0.0408, *w*R2 = 0.0721
R indices (all data)	R1 = 0.0995, *w*R2 = 0.0806
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.009
Average shift/error	0.000
Largest diff. peak and hole	3.252 and -2.305 e.Å^{-3}

Special Refinement Details

The disordered solvent region was modeled as two acetone and one water.

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.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C17
C19
Dy
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N5
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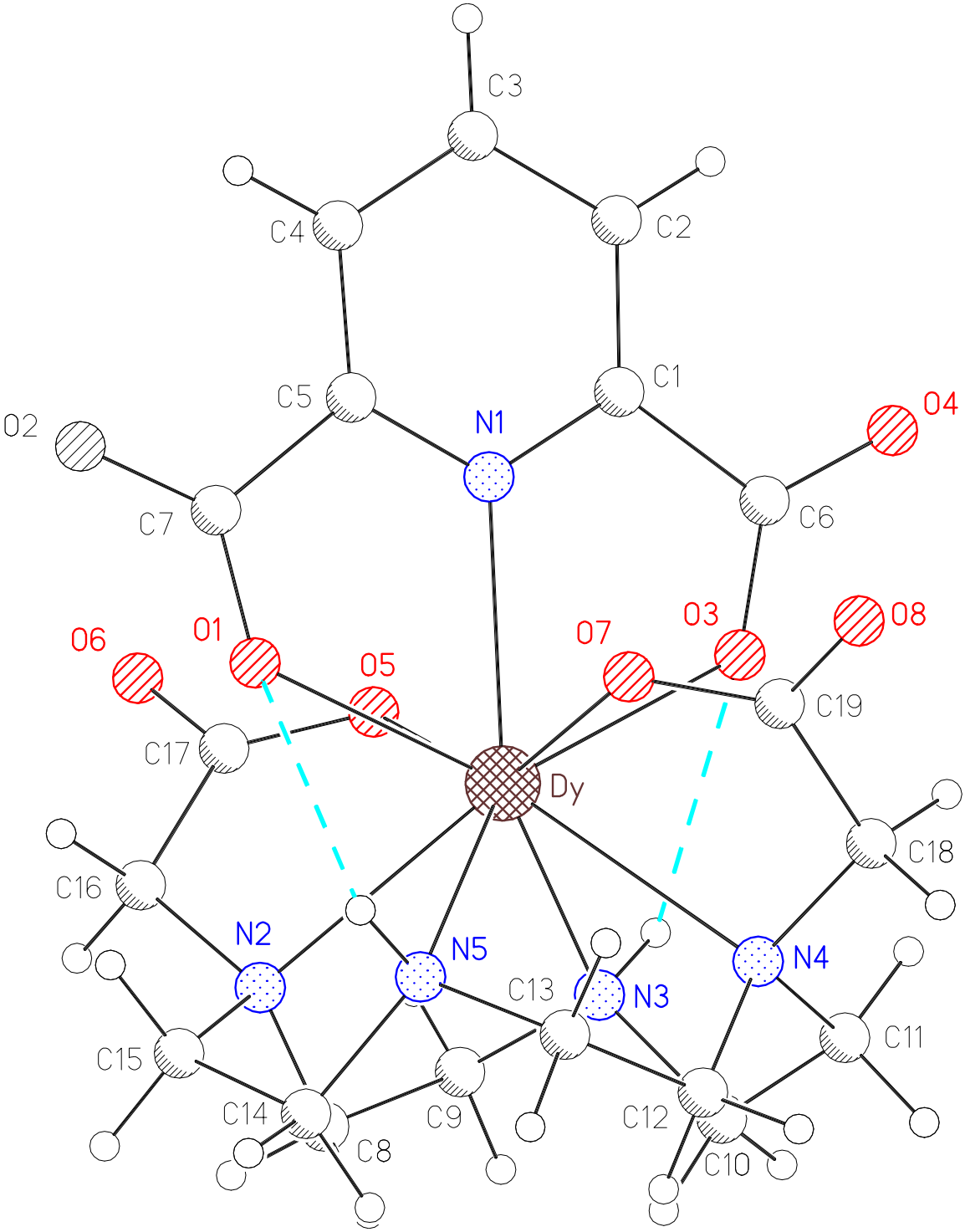
C8
C13
C14
C12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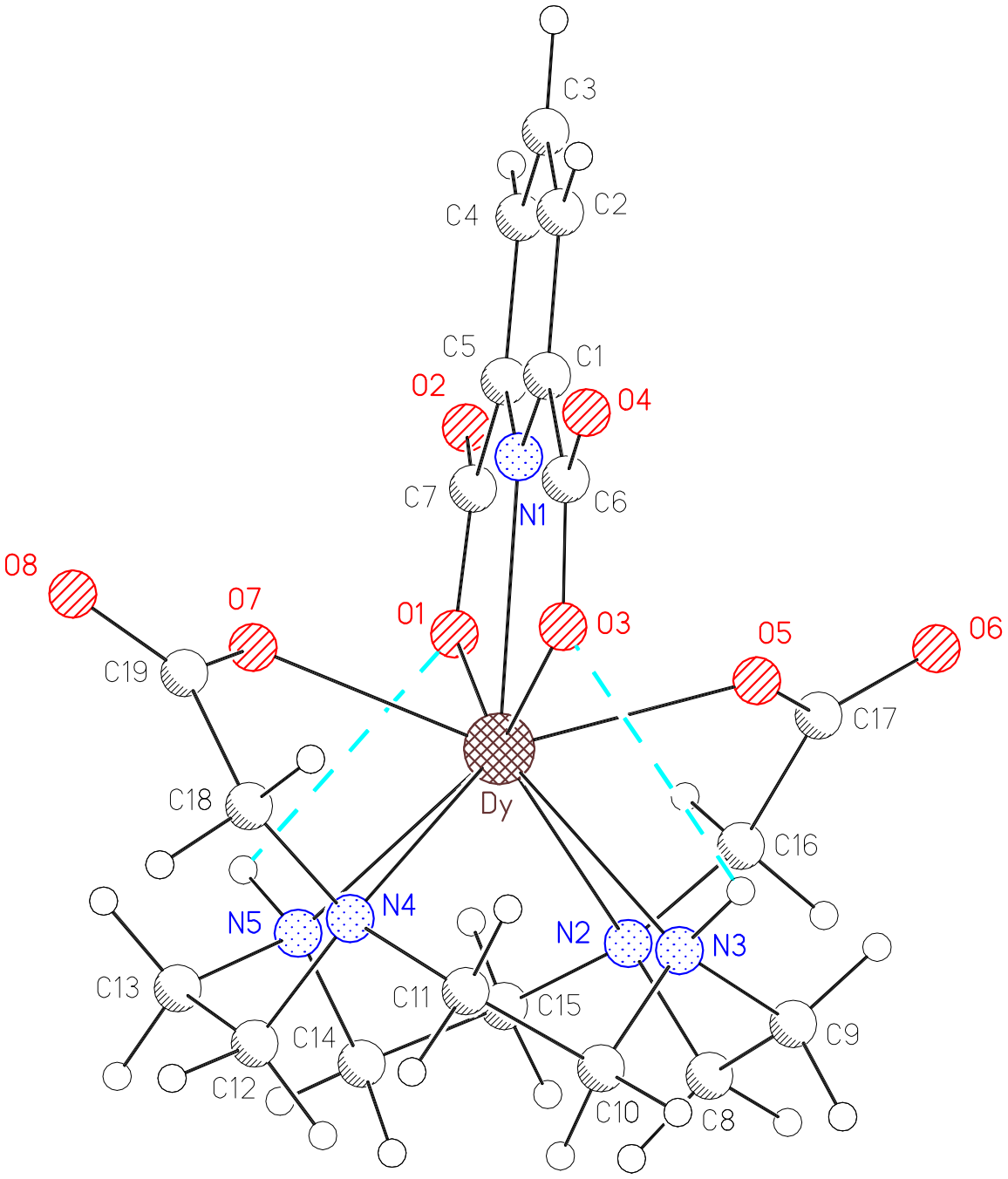
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C6
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O7
O1
O3
O5
O6
C19
C17
C18
Dy
C16
N5
N4
N2
N3
C13
C11
C15
C9
C14
C12
C10
C8

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for MLC06 (CCDC 643596). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}	Occ
Dy	2482(1)	52(1)	4985(1)	13(1)	1
O(1)	1700(1)	-860(1)	5658(1)	18(1)	1
O(2)	1398(1)	-2314(1)	6082(1)	28(1)	1
O(3)	3299(1)	-554(1)	4233(1)	18(1)	1
O(4)	3669(2)	-1796(1)	3679(1)	32(1)	1
O(5)	3923(1)	-581(1)	5402(1)	17(1)	1
O(6)	4664(1)	-1044(1)	6137(1)	18(1)	1
O(7)	1061(1)	-396(1)	4497(1)	17(1)	1
O(8)	278(1)	-479(1)	3738(1)	24(1)	1
N(1)	2499(1)	-1818(1)	4889(1)	15(1)	1
N(2)	2945(1)	1039(1)	5836(1)	18(1)	1
N(3)	3905(1)	1341(1)	4839(1)	18(1)	1
N(4)	2017(1)	1390(1)	4267(1)	19(1)	1
N(5)	1062(1)	1229(1)	5270(1)	18(1)	1
C(1)	2900(2)	-2236(2)	4468(1)	18(1)	1
C(2)	2933(2)	-3279(2)	4400(1)	28(1)	1
C(3)	2551(2)	-3901(2)	4787(1)	33(1)	1
C(4)	2156(2)	-3459(2)	5224(1)	28(1)	1
C(5)	2131(2)	-2403(1)	5261(1)	18(1)	1
C(6)	3319(2)	-1469(2)	4089(1)	19(1)	1
C(7)	1708(2)	-1822(2)	5709(1)	19(1)	1
C(8)	3637(2)	1899(2)	5730(1)	21(1)	1
C(9)	4421(2)	1650(2)	5320(1)	20(1)	1
C(10)	3647(2)	2216(2)	4511(1)	22(1)	1
C(11)	2958(2)	1879(2)	4072(1)	23(1)	1
C(12)	1324(2)	2174(2)	4468(1)	24(1)	1
C(13)	548(2)	1740(2)	4835(1)	21(1)	1
C(14)	1310(2)	1939(2)	5691(1)	21(1)	1
C(15)	1999(2)	1417(2)	6078(1)	20(1)	1
C(16)	3442(2)	282(2)	6174(1)	19(1)	1
C(17)	4067(2)	-506(1)	5881(1)	15(1)	1
C(18)	1525(2)	808(2)	3849(1)	22(1)	1
C(19)	892(2)	-86(2)	4041(1)	18(1)	1
N(6)	6380(1)	2787(1)	2784(1)	18(1)	1
C(20)	7199(2)	3421(2)	2523(1)	22(1)	1
C(21)	8262(2)	3278(2)	2740(1)	26(1)	1
C(22)	8998(2)	4000(2)	2480(1)	43(1)	1
C(23)	10095(2)	3775(3)	2645(1)	50(1)	1
C(24)	6671(2)	1667(1)	2784(1)	19(1)	1
C(25)	6818(2)	1205(2)	2258(1)	28(1)	1
C(26)	7082(2)	90(2)	2304(1)	28(1)	1
C(27)	7269(2)	-403(2)	1785(1)	36(1)	1
C(28)	5401(2)	2948(2)	2482(1)	21(1)	1
C(29)	4478(2)	2390(2)	2684(1)	24(1)	1
C(30)	3646(2)	2345(2)	2273(1)	26(1)	1
C(31)	2688(2)	1817(2)	2460(1)	36(1)	1

C(32)	6254(2)	3092(1)	3339(1)	19(1)	1
C(33)	6040(2)	4211(2)	3433(1)	26(1)	1
C(34)	5666(2)	4370(2)	3973(1)	30(1)	1
C(35)	5617(3)	5479(2)	4128(1)	50(1)	1
O(11)	5754(1)	4997(1)	1877(1)	26(1)	1
O(12)	9152(2)	909(1)	3124(1)	34(1)	1
O(51A)	1469(2)	4414(3)	6177(2)	66(2)	0.747(4)
C(52A)	552(2)	4385(2)	6237(1)	54(1)	0.747(4)
C(53A)	-5(3)	3688(3)	6616(2)	58(1)	0.747(4)
C(54A)	-53(3)	5123(5)	5895(2)	109(4)	0.747(4)
O(50A)	-1170(50)	2500(50)	6430(30)	1940(70)	0.747(4)
O(51B)	1223(8)	4340(6)	5350(3)	61(3)	0.253(4)
C(52B)	765(6)	4521(6)	5744(3)	42(3)	0.253(4)
C(53B)	-371(7)	4792(16)	5766(5)	102(10)	0.253(4)
C(54B)	1399(9)	4464(15)	6241(3)	75(10)	0.253(4)
O(50B)	-58(8)	3751(8)	7120(4)	61(3)	0.253(4)

Table 3. Selected bond lengths [Å] and angles [°] for MLC06 (CCDC 643596).

Dy-O(7)	2.3333(14)	O(7)-Dy-O(5)	144.16(5)
Dy-O(5)	2.3370(14)	O(7)-Dy-O(1)	85.70(5)
Dy-O(1)	2.3708(15)	O(5)-Dy-O(1)	80.10(5)
Dy-O(3)	2.3817(15)	O(7)-Dy-O(3)	80.27(5)
Dy-N(1)	2.4786(15)	O(5)-Dy-O(3)	83.97(5)
Dy-N(5)	2.5458(18)	O(1)-Dy-O(3)	129.90(6)
Dy-N(3)	2.5602(18)	O(7)-Dy-N(1)	72.59(5)
Dy-N(4)	2.6436(17)	O(5)-Dy-N(1)	71.58(5)
Dy-N(2)	2.6452(17)	O(1)-Dy-N(1)	64.81(5)
		O(3)-Dy-N(1)	65.09(5)
		O(7)-Dy-N(5)	74.04(5)
		O(5)-Dy-N(5)	132.63(5)
		O(1)-Dy-N(5)	76.62(6)
		O(3)-Dy-N(5)	141.46(5)
		N(1)-Dy-N(5)	130.01(6)
		O(7)-Dy-N(3)	132.31(5)
		O(5)-Dy-N(3)	73.32(5)
		O(1)-Dy-N(3)	140.22(5)
		O(3)-Dy-N(3)	76.45(6)
		N(1)-Dy-N(3)	129.67(6)
		N(5)-Dy-N(3)	100.30(6)
		O(7)-Dy-N(4)	66.48(5)
		O(5)-Dy-N(4)	138.86(6)
		O(1)-Dy-N(4)	139.87(5)
		O(3)-Dy-N(4)	75.15(5)
		N(1)-Dy-N(4)	126.51(5)
		N(5)-Dy-N(4)	68.47(6)
		N(3)-Dy-N(4)	67.56(5)
		O(7)-Dy-N(2)	139.74(6)
		O(5)-Dy-N(2)	66.48(5)
		O(1)-Dy-N(2)	74.03(5)
		O(3)-Dy-N(2)	139.02(5)
		N(1)-Dy-N(2)	124.89(5)
		N(5)-Dy-N(2)	67.67(5)
		N(3)-Dy-N(2)	68.46(6)
		N(4)-Dy-N(2)	108.60(6)

Table 4. Bond lengths [Å] and angles [°] for MLC06 (CCDC 643596).

Dy-O(7)	2.3333(14)	C(26)-C(27)	1.525(3)
Dy-O(5)	2.3370(14)	C(28)-C(29)	1.519(3)
Dy-O(1)	2.3708(15)	C(29)-C(30)	1.531(3)
Dy-O(3)	2.3817(15)	C(30)-C(31)	1.526(4)
Dy-N(1)	2.4786(15)	C(32)-C(33)	1.523(3)
Dy-N(5)	2.5458(18)	C(33)-C(34)	1.510(4)
Dy-N(3)	2.5602(18)	C(34)-C(35)	1.519(4)
Dy-N(4)	2.6436(17)	O(51A)-C(52A)	1.2200
Dy-N(2)	2.6452(17)	C(52A)-C(53A)	1.5400
O(1)-C(7)	1.275(2)	C(52A)-C(54A)	1.5400
O(2)-C(7)	1.243(3)	O(51B)-C(52B)	1.2200
O(3)-C(6)	1.264(2)	C(52B)-C(53B)	1.5400
O(4)-C(6)	1.244(3)	C(52B)-C(54B)	1.5400
O(5)-C(17)	1.267(2)		
O(6)-C(17)	1.249(2)	O(7)-Dy-O(5)	144.16(5)
O(7)-C(19)	1.277(2)	O(7)-Dy-O(1)	85.70(5)
O(8)-C(19)	1.242(2)	O(5)-Dy-O(1)	80.10(5)
N(1)-C(5)	1.333(3)	O(7)-Dy-O(3)	80.27(5)
N(1)-C(1)	1.341(3)	O(5)-Dy-O(3)	83.97(5)
N(2)-C(16)	1.481(3)	O(1)-Dy-O(3)	129.90(6)
N(2)-C(8)	1.483(3)	O(7)-Dy-N(1)	72.59(5)
N(2)-C(15)	1.487(3)	O(5)-Dy-N(1)	71.58(5)
N(3)-C(10)	1.476(3)	O(1)-Dy-N(1)	64.81(5)
N(3)-C(9)	1.480(3)	O(3)-Dy-N(1)	65.09(5)
N(4)-C(12)	1.478(3)	O(7)-Dy-N(5)	74.04(5)
N(4)-C(18)	1.481(3)	O(5)-Dy-N(5)	132.63(5)
N(4)-C(11)	1.489(3)	O(1)-Dy-N(5)	76.62(6)
N(5)-C(14)	1.479(3)	O(3)-Dy-N(5)	141.46(5)
N(5)-C(13)	1.481(3)	N(1)-Dy-N(5)	130.01(6)
C(1)-C(2)	1.388(3)	O(7)-Dy-N(3)	132.31(5)
C(1)-C(6)	1.522(3)	O(5)-Dy-N(3)	73.32(5)
C(2)-C(3)	1.397(4)	O(1)-Dy-N(3)	140.22(5)
C(3)-C(4)	1.388(4)	O(3)-Dy-N(3)	76.45(6)
C(4)-C(5)	1.396(3)	N(1)-Dy-N(3)	129.67(6)
C(5)-C(7)	1.509(3)	N(5)-Dy-N(3)	100.30(6)
C(8)-C(9)	1.528(3)	O(7)-Dy-N(4)	66.48(5)
C(10)-C(11)	1.524(3)	O(5)-Dy-N(4)	138.86(6)
C(12)-C(13)	1.516(3)	O(1)-Dy-N(4)	139.87(5)
C(14)-C(15)	1.519(3)	O(3)-Dy-N(4)	75.15(5)
C(16)-C(17)	1.533(3)	N(1)-Dy-N(4)	126.51(5)
C(18)-C(19)	1.530(3)	N(5)-Dy-N(4)	68.47(6)
N(6)-C(32)	1.514(3)	N(3)-Dy-N(4)	67.56(5)
N(6)-C(24)	1.526(2)	O(7)-Dy-N(2)	139.74(6)
N(6)-C(28)	1.522(3)	O(5)-Dy-N(2)	66.48(5)
N(6)-C(20)	1.528(3)	O(1)-Dy-N(2)	74.03(5)
C(20)-C(21)	1.519(3)	O(3)-Dy-N(2)	139.02(5)
C(21)-C(22)	1.522(3)	N(1)-Dy-N(2)	124.89(5)
C(22)-C(23)	1.534(4)	N(5)-Dy-N(2)	67.67(5)
C(24)-C(25)	1.515(3)	N(3)-Dy-N(2)	68.46(6)
C(25)-C(26)	1.516(3)	N(4)-Dy-N(2)	108.60(6)

C(7)-O(1)-Dy	125.37(14)
C(6)-O(3)-Dy	125.23(14)
C(17)-O(5)-Dy	123.25(13)
C(19)-O(7)-Dy	124.15(13)
C(5)-N(1)-C(1)	120.45(18)
C(5)-N(1)-Dy	119.89(13)
C(1)-N(1)-Dy	119.65(13)
C(16)-N(2)-C(8)	110.95(16)
C(16)-N(2)-C(15)	110.00(17)
C(8)-N(2)-C(15)	109.94(16)
C(16)-N(2)-Dy	105.52(11)
C(8)-N(2)-Dy	110.95(13)
C(15)-N(2)-Dy	109.39(12)
C(10)-N(3)-C(9)	112.28(16)
C(10)-N(3)-Dy	116.04(13)
C(9)-N(3)-Dy	112.98(13)
C(12)-N(4)-C(18)	110.86(17)
C(12)-N(4)-C(11)	109.69(17)
C(18)-N(4)-C(11)	109.57(18)
C(12)-N(4)-Dy	110.79(13)
C(18)-N(4)-Dy	105.93(12)
C(11)-N(4)-Dy	109.94(12)
C(14)-N(5)-C(13)	112.28(16)
C(14)-N(5)-Dy	116.38(13)
C(13)-N(5)-Dy	112.75(14)
N(1)-C(1)-C(2)	121.6(2)
N(1)-C(1)-C(6)	114.04(17)
C(2)-C(1)-C(6)	124.3(2)
C(1)-C(2)-C(3)	118.5(2)
C(4)-C(3)-C(2)	119.2(2)
C(3)-C(4)-C(5)	118.9(2)
N(1)-C(5)-C(4)	121.2(2)
N(1)-C(5)-C(7)	114.14(17)
C(4)-C(5)-C(7)	124.6(2)
O(4)-C(6)-O(3)	126.6(2)
O(4)-C(6)-C(1)	117.84(19)
O(3)-C(6)-C(1)	115.54(19)
O(2)-C(7)-O(1)	126.8(2)
O(2)-C(7)-C(5)	117.94(19)
O(1)-C(7)-C(5)	115.25(18)
N(2)-C(8)-C(9)	112.71(17)
N(3)-C(9)-C(8)	110.15(18)
N(3)-C(10)-C(11)	110.04(17)
N(4)-C(11)-C(10)	111.27(19)
N(4)-C(12)-C(13)	112.47(17)
N(5)-C(13)-C(12)	110.43(18)
N(5)-C(14)-C(15)	109.63(16)
N(2)-C(15)-C(14)	111.69(18)
N(2)-C(16)-C(17)	113.33(17)
O(6)-C(17)-O(5)	124.8(2)
O(6)-C(17)-C(16)	117.21(17)
O(5)-C(17)-C(16)	117.91(16)
N(4)-C(18)-C(19)	113.25(17)
O(8)-C(19)-O(7)	124.7(2)
O(8)-C(19)-C(18)	117.81(18)
O(7)-C(19)-C(18)	117.47(17)
C(32)-N(6)-C(24)	106.59(15)
C(32)-N(6)-C(28)	111.09(17)
C(24)-N(6)-C(28)	110.34(16)
C(32)-N(6)-C(20)	111.30(16)
C(24)-N(6)-C(20)	110.60(17)
C(28)-N(6)-C(20)	106.96(15)
C(21)-C(20)-N(6)	114.65(16)
C(20)-C(21)-C(22)	110.15(18)
C(21)-C(22)-C(23)	110.9(2)
C(25)-C(24)-N(6)	115.00(17)
C(26)-C(25)-C(24)	110.45(18)
C(25)-C(26)-C(27)	112.5(2)
C(29)-C(28)-N(6)	115.51(16)
C(28)-C(29)-C(30)	110.20(19)
C(31)-C(30)-C(29)	112.4(2)
N(6)-C(32)-C(33)	115.72(17)
C(34)-C(33)-C(32)	110.34(18)
C(33)-C(34)-C(35)	113.4(2)
O(51A)-C(52A)-C(53A)	125.2
O(51A)-C(52A)-C(54A)	114.6
C(53A)-C(52A)-C(54A)	120.2
O(51B)-C(52B)-C(53B)	124.2
O(51B)-C(52B)-C(54B)	115.6
C(53B)-C(52B)-C(54B)	120.2

Table 5. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for MLC06 (CCDC 643596). The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Dy	111(1)	169(1)	114(1)	13(1)	-13(1)	3(1)
O(1)	184(8)	225(7)	123(7)	34(5)	14(5)	0(5)
O(2)	363(10)	283(8)	201(8)	113(6)	67(7)	41(7)
O(3)	186(8)	236(7)	111(7)	1(5)	-7(5)	-9(5)
O(4)	477(12)	321(8)	154(8)	-80(6)	74(8)	-57(8)
O(5)	142(7)	241(7)	129(7)	-3(5)	-47(5)	26(5)
O(6)	180(7)	191(6)	160(7)	33(5)	-43(6)	-9(5)
O(7)	132(7)	250(7)	137(7)	26(5)	-27(5)	3(5)
O(8)	250(9)	304(8)	162(7)	-16(6)	-68(6)	26(6)
N(1)	122(7)	187(6)	128(8)	6(5)	-32(7)	14(5)
N(2)	154(9)	215(8)	172(8)	-7(6)	-15(7)	6(6)
N(3)	154(9)	193(7)	177(9)	26(6)	-2(7)	-4(6)
N(4)	152(9)	238(8)	175(9)	49(6)	-11(7)	5(6)
N(5)	178(9)	176(7)	195(9)	5(6)	-22(7)	12(6)
C(1)	146(10)	234(9)	163(10)	-23(7)	-34(7)	-6(7)
C(2)	271(13)	243(10)	310(13)	-82(8)	-1(10)	-5(8)
C(3)	363(15)	211(10)	419(15)	-8(9)	78(12)	-17(9)
C(4)	268(13)	222(10)	349(14)	50(9)	48(10)	9(8)
C(5)	151(10)	190(8)	195(10)	48(7)	-27(7)	17(6)
C(6)	164(10)	277(9)	131(9)	-26(7)	-41(8)	-9(7)
C(7)	144(10)	247(9)	166(10)	58(7)	-11(8)	15(7)
C(8)	211(11)	203(8)	216(11)	-44(7)	-14(8)	-16(7)
C(9)	159(10)	229(9)	219(11)	-14(7)	-26(8)	-38(7)
C(10)	199(11)	225(9)	248(11)	61(8)	3(9)	-35(7)
C(11)	216(11)	245(9)	229(11)	91(8)	6(9)	-20(8)
C(12)	220(11)	245(9)	247(12)	78(8)	-20(9)	52(8)
C(13)	165(10)	223(9)	230(11)	32(7)	-21(8)	39(7)
C(14)	170(11)	231(9)	228(11)	-35(8)	-10(8)	38(7)
C(15)	192(11)	245(9)	169(10)	-50(7)	-7(8)	20(7)
C(16)	199(10)	238(8)	134(9)	-20(6)	-45(7)	21(7)
C(17)	137(9)	185(8)	137(9)	17(6)	-21(7)	-32(6)
C(18)	203(11)	305(10)	164(10)	65(8)	-31(8)	-15(8)
C(19)	139(8)	254(9)	131(8)	-2(7)	-20(6)	68(7)
N(6)	160(9)	214(7)	167(9)	68(6)	-17(7)	5(6)
C(20)	187(11)	266(9)	197(11)	125(8)	21(8)	0(7)
C(21)	192(11)	340(11)	262(12)	139(9)	0(9)	-7(8)
C(22)	199(13)	557(17)	525(19)	336(14)	4(12)	-26(11)
C(23)	214(14)	608(19)	680(20)	299(17)	-30(14)	-84(12)
C(24)	199(10)	195(8)	179(10)	65(7)	0(8)	21(7)
C(25)	352(14)	275(10)	203(11)	32(8)	30(10)	54(9)
C(26)	322(12)	250(10)	272(11)	-7(9)	-32(9)	48(9)
C(27)	354(15)	385(13)	334(15)	-79(11)	-3(12)	77(11)
C(28)	180(10)	247(9)	189(10)	86(7)	-26(8)	29(7)
C(29)	178(11)	343(11)	196(11)	74(8)	-23(8)	-7(8)
C(30)	265(13)	288(10)	229(12)	34(8)	-64(10)	-20(8)
C(31)	245(14)	404(13)	431(17)	68(11)	-108(12)	-82(10)

C(32)	196(10)	200(8)	168(9)	57(7)	-7(8)	-4(7)
C(33)	290(13)	222(9)	259(12)	42(8)	34(9)	13(8)
C(34)	368(15)	241(10)	299(14)	-42(9)	22(11)	3(9)
C(35)	590(20)	342(15)	570(20)	-136(14)	145(17)	12(13)
O(11)	358(9)	240(7)	178(7)	14(6)	-72(6)	62(7)
O(12)	392(11)	400(10)	227(9)	9(7)	-97(8)	141(8)
O(51A)	380(30)	520(20)	1090(40)	-80(20)	10(30)	7(19)
C(52A)	360(30)	660(30)	600(30)	-230(20)	20(20)	90(20)
C(53A)	420(30)	610(30)	700(40)	-170(20)	-10(20)	50(20)
C(54A)	310(40)	2180(110)	800(50)	580(60)	40(30)	260(50)
O(51B)	1020(90)	270(40)	540(60)	80(40)	250(60)	-40(40)
C(52B)	730(90)	180(40)	360(60)	90(40)	80(60)	-90(40)
C(53B)	180(90)	1900(300)	1000(180)	360(170)	-120(90)	-10(120)
C(54B)	280(100)	1800(300)	130(60)	-250(90)	30(60)	-190(110)

Table 6. Hydrogen bonds for MLC06 (CCDC 643596) [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(3)-H(3)...O(5)#1	0.93	2.29	3.101(2)	146.1
N(3)-H(3)...O(6)#1	0.93	2.42	3.204(3)	141.4
N(5)-H(5)...O(7)#2	0.93	2.23	3.069(2)	149.4
N(5)-H(5)...O(8)#2	0.93	2.54	3.297(3)	138.2
O(11)-H(11A)...O(4)#3	0.99	1.89	2.879(2)	175.2
O(11)-H(11A)...O(3)#3	0.99	2.55	3.246(2)	126.5
O(11)-H(11B)...O(6)#4	0.99	1.83	2.7691(19)	155.9
O(12)-H(12A)...O(2)#1	1.00	1.89	2.875(3)	170.7
O(12)-H(12B)...O(8)#5	1.00	1.88	2.844(2)	160.8

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