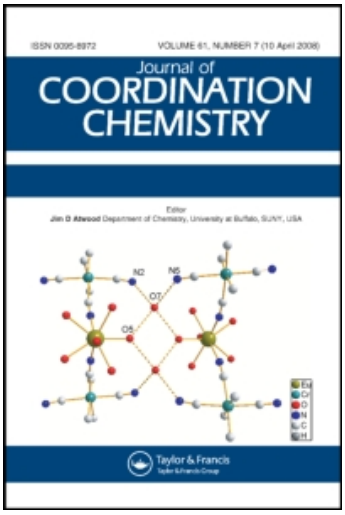

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STUDIES OF LANTHANOID COMPLEXES WITH CROWN ETHERS XXVI. SYNTHESIS AND STRUCTURE OF THE 3:2 COMPLEX OF YTTRIUM NITRATE WITH DIBENZO-30-CROWN-10

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STUDIES OF LANTHANOID COMPLEXES WITH CROWN ETHERS XXVI. SYNTHESIS AND STRUCTURE OF THE 3:2 COMPLEX OF YTTRIUM NITRATE WITH DIBENZO-30-CROWN-10

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The new compound $[Y(NO_3)_2(OH_2)_2(DB30C10)]_2[Y(NO_3)_5].5CH_3NO_2$ has been synthesized and its crystal and molecular structure determined using single crystal X-ray techniques. The colourless, air-sensitive crystals belong to the triclinic space group $P\bar{1}$ with $Z=2$. Lattice parameters are $a=11.218(2)$, $b=18.530(4)$, $c=25.758(5)$Å, $\alpha=70.96(1)$, $\beta=89.36(1)$, $\gamma=73.92(1)°$, $V=4845(2)$Å^3. The structure was solved and refined to $R=0.059$ for 4494 independent reflections with $I\geqslant 3\sigma(I)$. The asymmetric unit contains three independent ions: two $[Y(NO_3)_2(OH_2)_2(DB30C10)]^+$ cations, in which the coordination number of Y^{3+} is nine, and one $[Y(NO_3)_5]^{2-}$ anion, in which the coordination number of Y^{3+} is ten.

KEY WORDS: Yttrium nitrate, dibenzo-30-crown-10, complex, crystal structure.

INTRODUCTION

Many complexes of crown ethers with rare earth ions have been synthesized and characterized over recent years.[1] However, complexes of crown ethers which contain more than eight ether oxygen atoms have seldom been reported.[2] In order to investigate the coordination of crown ethers containing more than eight ether oxygen atoms with rare earth ions, we recently synthesized a series of large cavity crown ethers with rare earths. The present paper reports the synthesis and crystal structure of a complex of yttrium(III) nitrate with DB30C10.

EXPERIMENTAL

Chemicals

Hydrated yttrium nitrate was prepared by dissolving Y_2O_3 (AnalaR) in 1:1 nitric acid.

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The ligand was obtained from the Tuejiang chemical factory in Sichuan province. All other chemicals were AnalaR grade.

Preparation

A solution of 0.4 mmol $Y(NO_3)_3.nH_2O$ in 3 cm^3 of acetonitrile was added dropwise to a solution of 0.2 mmol of DB30C10 in 3 cm^3 of acetonitrile. The mixture was stirred at room temperature for 12 h. The precipitated solid complex was filtered, washed with acetonitrile and then the solid complex was dissolved in nitromethane. The solution was placed in a desiccator over $CaCl_2$ at room temperature. After about one month, colourless, air-sensitive crystals of the title complex had formed from the solution in 80% yield. Anal: calcd. for $C_{61}H_{103}N_{14}O_{61}Y_3$: C, 32.20; H, 4.78; N, 8.62; Y, 11.72%; found: C, 32.58; H, 4.97; N, 8.33; Y, 12.05%.

Crystal Structure Analysis

X-ray diffraction intensities were collected at 25°C on an R3M/E diffractometer with graphite-monochromated KoK_α radiation, using the $\theta/2\theta$ scan technique. The reflections were corrected for absorption by the Gaussian integration method, for Lorentz polarization and secondary extinction effects. Crystal data are listed in Table 1.

The structure was solved by the Patterson method and subsequent difference Fourier techniques and refined by full-matrix least-squares methods with anisotropic thermal factors for all non-hydrogen atoms; a number of the hydrogen atoms were

Table 1 Crystal data

Formula		$C_{61}H_{103}N_{14}O_{61}Y_3$
Crystal dimension, mm		0.32 × 0.40 × 0.60
System		triclinic
Space group		$P\bar{1}$
Lattice parameters	*a*, Å	11.218(2)
	b, Å	18.530(4)
	c, Å	25.758(5)
	α, deg	70.96(1)
	β, deg	89.36(1)
	γ, deg	73.92(1)
Vol. (Å^3)		4845(2)
Z		2
D_c (g cm^{-3})		1.56
D_o (g cm^{-3})		1.59
μ(calc), cm^{-1}		19.01
F(000)		2344
Scan rate		10°/min
Standard reflection		2,0,2,2,−3,0
2θ range, deg		1–43
Unique data measured		11924
Observable data with $I \geqslant 3.0\sigma(I)$		4494
No. of variables		1253
Octants		*h*,*k*,*l*(0 to 12, −20 to 20, −27 to 27)
S		1.086
R		0.0590
R_w		0.0526

placed in calculated positions. All calculations were performed using the SHELXTL system of computer programs. The final values $R = 0.059$, $R_w = 0.053$ were obtained.

RESULT AND DISCUSSION

Final atomic positional and thermal parameters are given in Table 2 and selected bond lengths and bond angles in Tables 3 and 4. Figure 1 shows the structure of the $[Y_{(1)}(NO_3)_2(OH_2)_2(DB30C10)]^+$ and $[Y_{(2)}(NO_3)_2(OH_2)_2(DB30C10)]^+$ cations. Figure 2 shows the structure of the $[Y_{(3)}(NO_3)_5]^{2-}$ anion.

The structure contains two independent $[Y(NO_3)_2(OH_2)_2(DB30C10)]^+$ cations, one $[Y(NO_3)_5]^{2-}$ anion and five uncoordinated nitromethane molecules. In the cations, the nine-coordinate Y^{3+} ions are coordinated to three crown ether oxygen

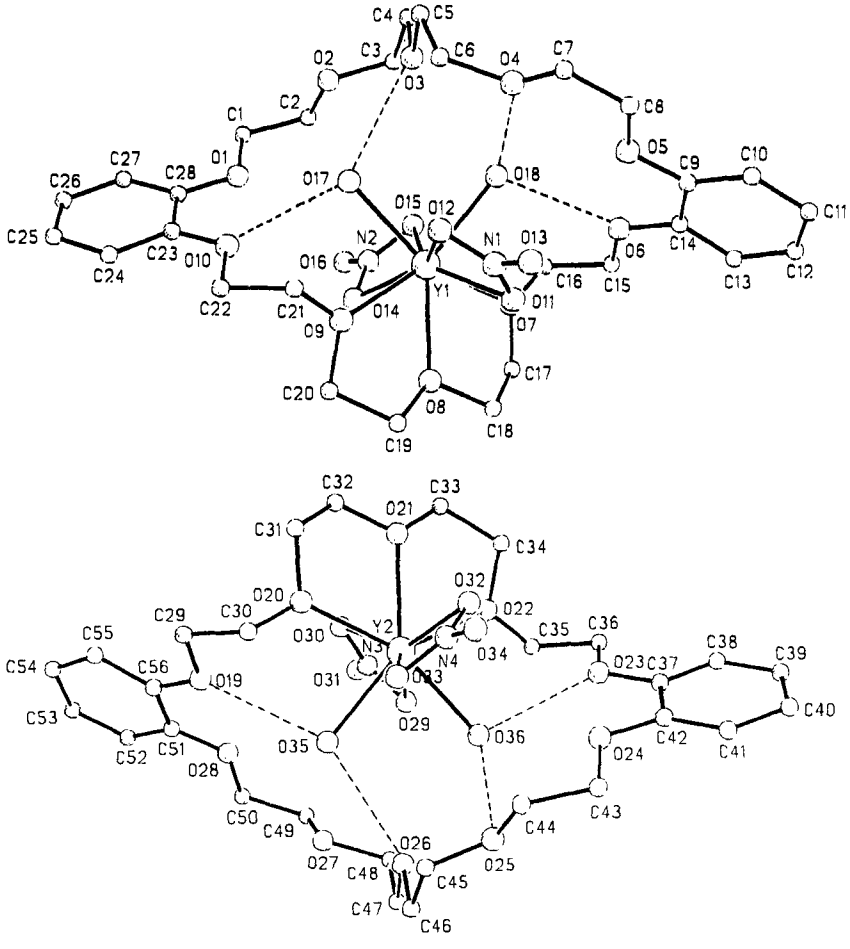

Figure 1 The structures of the $[Y(NO_3)_2(OH_2)_2(DB30C10)]^+$ cations.

Table 2 Final atomic positional ($\times 10^4$) and equivalent isotropic thermal parameters ($Å^2 \times 10^3$)

	x/a	*y/b*	*z/c*	*Ueq**
Y(1)	3851(1)	1467(1)	1110(1)	43(1)
Y(2)	9507(1)	1543(1)	6016(1)	44(1)
Y(3)	7190(2)	4646(1)	7569(1)	76(1)
N(1)	5725(9)	1498(6)	1824(4)	62(5)
N(2)	1999(10)	1445(6)	362(4)	65(5)
N(3)	7592(9)	1692(6)	6747(4)	61(5)
N(4)	11450(10)	1490(6)	5290(4)	73(6)
N(5)	6717(12)	3107(7)	7982(5)	104(7)
N(6)	7696(17)	6132(8)	7158(6)	164(10)
N(7)	9780(11)	3869(9)	7694(5)	120(8)
N(8)	5948(14)	5217(9)	8426(5)	134(9)
N(9)	5778(12)	4693(7)	6625(5)	113(7)
N(10)	7928(14)	2572(8)	2391(5)	119(9)
N(11)	3862(12)	2548(9)	7402(6)	128(9)
N(12)	6409(17)	928(7)	4544(5)	150(9)
N(13)	8128(33)	4696(25)	2485(13)	400(34)
N(14)	7821(16)	1096(19)	9583(7)	231(18)
O(1)	730(8)	3509(5)	−160(3)	80(5)
O(2)	2878(9)	3349(5)	−749(4)	86(5)
O(3)	5115(9)	3140(5)	−168(4)	90(5)
O(4)	7128(12)	2473(6)	641(7)	204(10)
O(5)	8323(7)	898(4)	1049(3)	67(4)
O(6)	7210(7)	−135(4)	1012(3)	61(4)
O(7)	4607(7)	87(4)	1166(3)	56(4)
O(8)	3083(7)	582(4)	1852(3)	66(4)
O(9)	2553(7)	2106(5)	1682(3)	58(4)
O(10)	899(7)	3474(5)	857(3)	68(4)
O(11)	5525(8)	852(4)	1839(3)	69(4)
O(12)	5024(7)	2133(4)	1503(3)	60(4)
O(13)	6604(8)	1485(5)	2113(3)	78(5)
O(14)	1817(7)	1503(5)	841(3)	68(4)
O(15)	3141(7)	1359(4)	246(3)	60(4)
O(16)	1196(9)	1440(6)	52(4)	103(6)
O(17)	3181(7)	2779(4)	500(3)	56(4)
O(18)	5613(7)	1471(4)	610(3)	55(4)
O(19)	7673(7)	−138(4)	6032(3)	64(4)
O(20)	10007(7)	134(4)	6126(3)	58(4)
O(21)	11060(7)	672(4)	6789(3)	64(4)
O(22)	10104(7)	2236(4)	6560(3)	54(4)
O(23)	10579(7)	3573(5)	5744(3)	68(4)
O(24)	10850(7)	3604(5)	4733(3)	65(4)
O(25)	8934(9)	3306(5)	4178(4)	92(5)
O(26)	6707(9)	3073(5)	4642(4)	99(5)
O(27)	5124(9)	2463(5)	5449(4)	115(6)
O(28)	5654(7)	883(5)	6105(3)	71(4)
O(29)	7632(7)	2285(4)	6325(3)	60(4)
O(30)	8395(7)	1027(4)	6792(3)	71(4)
O(31)	6778(8)	1763(5)	7063(4)	95(5)
O(32)	11529(7)	1596(5)	5750(3)	72(5)
O(33)	10424(7)	1366(5)	5178(3)	64(4)
O(34)	12265(8)	1505(6)	4976(4)	90(5)
O(35)	7774(7)	1494(4)	522(3)	60(4)
O(36)	9049(7)	2833(4)	5367(3)	57(4)
O(37)	7518(11)	3256(7)	7663(6)	158(8)
O(38)	6193(18)	3677(8)	8068(7)	233(12)
O(39)	6506(11)	2470(6)	8163(5)	153(7)

Table 2 *continued*

	x/a	*y/b*	*z/c*	*Ueq**
O(40)	8073(17)	5661(8)	7643(6)	199(11)
O(41)	7119(15)	5929(7)	6872(5)	173(9)
O(42)	7954(18)	6762(7)	6995(7)	221(12)
O(43)	9204(9)	4345(6)	7237(4)	109(6)
O(44)	9210(12)	3896(9)	8097(4)	166(9)
O(45)	10888(10)	3475(10)	7744(5)	174(9)
O(46)	7025(11)	4732(8)	8482(4)	161(8)
O(47)	5517(11)	5373(7)	7949(4)	129(7)
O(48)	5443(14)	5468(7)	8756(5)	179(9)
O(49)	5293(11)	4890(8)	7008(5)	149(8)
O(50)	6927(10)	4523(7)	6677(4)	122(6)
O(51)	5198(12)	4615(7)	6263(4)	139(7)
O(52)	8766(13)	2323(11)	2195(8)	230(14)
O(53)	7790(19)	2330(8)	2862(5)	224(12)
O(54)	3789(11)	2030(8)	7266(6)	153(9)
O(55)	3644(14)	2509(8)	7865(6)	178(9)
O(56)	6837(17)	1395(8)	4302(7)	205(11)
O(57)	6861(18)	268(9)	4699(7)	232(12)
O(58)	8663(20)	4211(14)	2341(11)	308(20)
O(59)	8236(20)	4821(11)	2890(9)	274(15)
O(60)	6826(11)	1241(9)	9512(6)	173(10)
O(61)	7892(22)	371(11)	9658(9)	342(19)
C(1)	661(14)	3586(9)	−743(5)	104(9)
C(2)	1887(13)	3069(8)	−835(5)	84(8)
C(3)	4058(14)	2881(9)	−852(6)	104(9)
C(4)	4978(14)	3251(8)	−727(6)	99(9)
C(5)	5868(18)	3569(10)	−50(7)	127(11)
C(6)	6351(21)	3301(11)	465(9)	199(16)
C(7)	8102(15)	2274(9)	793(11)	183(15)
C(8)	8870(12)	1489(7)	1087(6)	74(7)
C(9)	9018(12)	114(7)	1269(5)	55(6)
C(10)	10224(11)	−166(8)	1510(5)	70(7)
C(11)	10837(16)	−990(11)	1713(6)	93(9)
C(12)	10227(17)	−1500(11)	1685(7)	92(10)
C(13)	9037(14)	−1261(8)	1459(6)	76(8)
C(14)	8410(11)	−413(7)	1247(5)	60(6)
C(15)	6536(11)	−661(7)	942(5)	67(7)
C(16)	5234(11)	−208(7)	748(5)	66(7)
C(17)	4067(13)	−468(7)	1549(5)	78(7)
C(18)	3831(13)	−239(7)	2047(5)	78(8)
C(19)	2647(13)	833(8)	2303(5)	82(8)
C(20)	1848(12)	1669(8)	2063(5)	82(8)
C(21)	2334(11)	2937(8)	1650(5)	73(7)
C(22)	1048(13)	3461(8)	1399(5)	85(8)
C(23)	−155(11)	3967(6)	540(5)	68(7)
C(24)	−1100(12)	4439(7)	744(7)	94(8)
C(25)	−2175(14)	4959(8)	406(7)	102(9)
C(26)	−2271(13)	4972(8)	−121(8)	117(10)
C(27)	−1340(12)	4497(7)	−345(6)	83(7)
C(28)	−265(12)	3982(7)	−4(5)	67(7)
C(29)	8835(11)	−712(7)	5977(5)	65(7)
C(30)	9684(11)	−207(7)	5722(5)	59(7)
C(31)	11060(15)	−438(8)	6514(6)	97(8)
C(32)	11087(13)	−152(6)	7002(5)	79(7)
C(33)	11301(12)	993(7)	7210(5)	70(7)
C(34)	11241(13)	1866(8)	6922(5)	78(7)
C(35)	9494(13)	3075(7)	6510(5)	75(7)

Table 2 *continued*

	x/a	*y/b*	*z/c*	*U*eq*
C(36)	10369(14)	3592(8)	6303(5)	78(8)
C(37)	11232(11)	4070(7)	5432(5)	57(6)
C(38)	11707(11)	4551(7)	5643(5)	68(7)
C(39)	12367(13)	5051(8)	5299(6)	90(8)
C(40)	12511(12)	5065(7)	4759(6)	88(8)
C(41)	12035(12)	4582(7)	4545(6)	74(7)
C(42)	11379(11)	4093(7)	4886(5)	56(6)
C(43)	10810(13)	3687(9)	4160(5)	82(8)
C(44)	10216(13)	3105(8)	4058(5)	77(8)
C(45)	8240(15)	2846(9)	4010(6)	95(9)
C(46)	6890(15)	3240(9)	4061(6)	105(9)
C(47)	5416(16)	3523(10)	4683(7)	135(10)
C(48)	5255(16)	3293(8)	5279(7)	129(10)
C(49)	4616(15)	2280(11)	5933(7)	116(11)
C(50)	4464(12)	1466(8)	6074(6)	86(7)
C(51)	5622(11)	123(7)	6297(5)	63(7)
C(52)	4609(13)	−158(9)	6520(5)	90(8)
C(53)	4715(14)	−955(9)	6730(6)	94(8)
C(54)	5828(14)	−1531(8)	6696(5)	83(8)
C(55)	6868(13)	−1266(8)	6454(5)	77(7)
C(56)	6738(12)	−457(8)	6264(4)	64(7)
C(57)	6852(19)	3216(10)	2042(9)	168(13)
C(58)	4215(21)	3261(9)	7057(8)	155(12)
C(59)	5092(16)	1224(18)	4704(9)	208(19)
C(60)	7290(28)	5270(16)	2063(12)	250(20)
C(61)	8761(18)	1314(16)	9622(10)	307(31)

*Equivalent isotropic U defined as one third of the trace of the orthogonalised U_{ij} tensor.

Table 3 Selected bond lengths (Å)

Y(1)-O(7)	2.418(7)	Y(2)-O(32)	2.381(8)
Y(1)-O(8)	2.405(8)	Y(2)-O(33)	2.459(8)
Y(1)-O(9)	2.412(8)	Y(2)-O(35)	2.374(9)
Y(1)-O(11)	2.417(8)	Y(2)-O(36)	2.347(6)
Y(1)-O(12)	2.455(9)	Y(3)-O(37)	2.429(13)
Y(1)-O(14)	2.369(9)	Y(3)-O(38)	2.388(18)
Y(1)-O(15)	2.459(9)	Y(3)-O(40)	2.414(19)
Y(1)-O(17)	2.338(6)	Y(3)-O(41)	2.450(12)
Y(1)-O(18)	2.349(8)	Y(3)-O(43)	2.415(10)
Y(2)-O(20)	2.434(8)	Y(3)-O(44)	2.451(11)
Y(2)-O(21)	2.455(7)	Y(3)-O(46)	2.409(13)
Y(2)-O(22)	2.396(9)	Y(3)-O(47)	2.389(12)
Y(2)-O(29)	2.454(8)	Y(3)-O(49)	2.436(13)
Y(2)-O(30)	2.413(8)	Y(3)-O(50)	2.410(11)

Table 4 Selected bond angles (deg)

O(7)-Y(1)-O(8)	66.6(3)	O(8)-Y(1)-O(15)	107.3(3)
O(7)-Y(1)-O(9)	132.0(3)	O(9)-Y(1)-O(15)	125.8(3)
O(8)-Y(1)-O(9)	65.5(3)	O(11)-Y(1)-O(15)	141.5(3)
O(7)-Y(1)-O(11)	74.3(3)	O(12)-Y(1)-O(15)	144.2(3)
O(8)-Y(1)-O(11)	74.4(3)	O(14)-Y(1)-O(15)	52.8(3)
O(9)-Y(1)-O(11)	90.7(3)	O(7)-Y(1)-O(17)	143.9(3)
O(7)-Y(1)-O(12)	124.0(2)	O(8)-Y(1)-O(17)	138.7(3)
O(8)-Y(1)-O(12)	108.5(3)	O(9)-Y(1)-O(17)	79.7(2)
O(9)-Y(1)-O(12)	70.7(3)	O(11)-Y(1)-O(17)	129.6(3)
O(11)-Y(1)-O(12)	52.4(3)	O(12)-Y(1)-O(17)	78.0(3)
O(7)-Y(1)-O(14)	90.0(3)	O(14)-Y(1)-O(17)	80.9(3)
O(8)-Y(1)-O(14)	70.3(3)	O(15)-Y(1)-O(17)	75.0(3)
O(9)-Y(1)-O(14)	76.5(3)	O(7)-Y(1)-O(18)	78.4(3)
O(11)-Y(1)-O(14)	144.7(3)	O(8)-Y(1)-O(18)	140.0(2)
O(12)-Y(1)-O(14)	143.5(3)	O(9)-Y(1)-O(18)	143.7(3)
O(7)-Y(1)-O(15)	71.7(2)	O(11)-Y(1)-O(18)	78.2(3)
O(12)-Y(1)-O(18)	75.2(3)	O(14)-Y(1)-O(18)	130.2(3)
O(15)-Y(1)-O(18)	77.7(3)	O(17)-Y(1)-O(18)	81.2(2)
O(11)-N(1)-O(12)	117.8(10)	O(14)-N(2)-O(15)	113.3(10)
O(20)-Y(2)-O(21)	66.1(3)		
O(20)-Y(2)-O(22)	132.4(2)	O(21)-Y(2)-O(22)	66.5(3)
O(20)-Y(2)-O(29)	124.3(3)	O(21)-Y(2)-O(29)	111.9(3)
O(22)-Y(2)-O(29)	71.0(3)	O(20)-Y(2)-O(30)	76.9(3)
O(21)-Y(2)-O(30)	72.5(3)	O(22)-Y(2)-O(30)	84.9(3)
O(29)-Y(2)-O(30)	52.9(2)	O(20)-Y(2)-O(32)	92.1(2)
O(21)-Y(2)-O(32)	70.8(2)	O(22)-Y(2)-O(32)	76.8(3)
O(29)-Y(2)-O(32)	142.2(3)	O(30)-Y(2)-O(32)	143.0(2)
O(20)-Y(2)-O(33)	72.2(3)	O(21)-Y(2)-O(33)	106.2(2)
O(22)-Y(2)-O(33)	126.5(3)	O(29)-Y(2)-O(33)	141.8(2)
O(30)-Y(2)-O(33)	146.2(3)	O(32)-Y(2)-O(33)	52.8(3)
O(20)-Y(2)-O(35)	77.6(3)	O(21)-Y(2)-O(35)	139.1(3)
O(22)-Y(2)-O(35)	142.7(3)	O(29)-Y(2)-O(35)	73.2(3)
O(30)-Y(2)-O(35)	81.7(3)	O(32)-Y(2)-O(35)	130.8(3)
O(33)-Y(2)-O(35)	78.6(3)	O(20)-Y(2)-O(36)	144.1(3)
O(21)-Y(2)-O(36)	138.4(3)	O(22)-Y(2)-O(36)	79.6(3)
O(29)-Y(2)-O(36)	76.4(2)	O(30)-Y(2)-O(36)	129.3(3)
O(32)-Y(2)-O(36)	78.9(3)	O(33)-Y(2)-O(36)	74.8(3)
O(35)-Y(2)-O(36)	82.4(3)	O(29)-N(3)-O(30)	115.7(9)
O(32)-N(2)-O(33)	114.6(9)		
O(37)-Y(3)-O(38)	47.5(5)	O(37)-Y(3)-O(40)	147.7(5)
O(38)-Y(3)-O(40)	143.5(6)	O(37)-Y(3)-O(41)	140.6(5)
O(38)-Y(3)-O(41)	151.0(6)	O(40)-Y(3)-O(41)	51.4(5)
O(37)-Y(3)-O(43)	75.0(4)	O(38)-Y(3)-O(43)	120.8(5)
O(40)-Y(3)-O(43)	79.3(4)	O(41)-Y(3)-O(43)	82.0(4)
O(37)-Y(3)-O(44)	73.0(5)	O(38)-Y(3)-O(44)	93.5(5)
O(40)-Y(3)-O(44)	75.7(5)	O(41)-Y(3)-O(44)	115.5(5)
O(43)-Y(3)-O(44)	51.0(4)	O(37)-Y(3)-O(46)	107.1(5)
O(38)-Y(3)-O(46)	73.5(6)	O(40)-Y(3)-O(46)	70.0(5)
O(41)-Y(3)-O(46)	112.2(5)	O(43)-Y(3)-O(46)	120.6(4)
O(44)-Y(3)-O(46)	72.4(4)	O(37)-Y(3)-O(47)	123.1(4)
O(38)-Y(3)-O(47)	75.8(5)	O(40)-Y(3)-O(47)	80.6(5)
O(41)-Y(3)-O(47)	86.7(4)	O(43)-Y(3)-O(47)	159.9(5)
O(44)-Y(3)-O(47)	121.7(4)	O(46)-Y(3)-O(47)	49.5(4)
O(37)-Y(3)-O(49)	85.1(5)	O(38)-Y(3)-O(49)	73.5(5)
O(40)-Y(3)-O(49)	125.3(5)	O(41)-Y(3)-O(49)	79.5(5)
O(43)-Y(3)-O(49)	120.4(4)	O(44)-Y(3)-O(49)	157.8(6)
O(46)-Y(3)-O(49)	118.9(4)	O(47)-Y(3)-O(49)	73.3(4)

Table 4 *continued*

O(37)-Y(3)-O(50)	69.9(4)	O(38)-Y(3)-O(50)	97.2(5)
O(40)-Y(3)-O(50)	119.0(4)	O(41)-Y(3)-O(50)	72.4(5)
O(43)-Y(3)-O(50)	70.4(4)	O(44)-Y(3)-O(50)	116.4(4)
O(46)-Y(3)-O(50)	168.0(4)	O(47)-Y(3)-O(50)	121.7(4)
O(49)-Y(3)-O(50)	50.0(4)	O(37)-N(5)-O(38)	108.8(15)
O(40)-N(6)-O(41)	118.1(7)	O(43)-N(7)-O(44)	117.2(12)
O(46)-N(8)-O(47)	106.6(13)	O(49)-N(9)-O(50)	112.3(14)

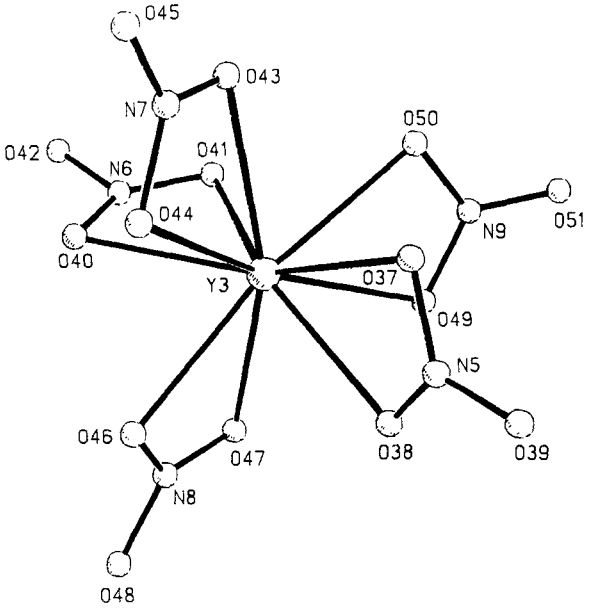

Figure 2 The structure of the $[Y_{(3)}(NO_3)_5]^{2-}$ anion.

atoms, two bidentate nitrate groups and two water molecules. The bond lengths lie in the range Y(1)-O(crown), 2.405–2.418Å; Y(1)-O(NO_3^-), 2.369–2.459Å; Y(1)-O(water), 2.338–2.349Å; Y(2)-O(crown), 2.396–2.455Å; Y(2)-O(NO_3^-), 2.381–2.459Å; Y(2)-O(water), 2.347–2.374Å. The coordination geometries of these two cations are distorted tricapped trigonal prisms[3] (Figure 3), similar to those observed in $[MCl(OH_2)_2(18\text{-crown-6})]^{2+}$ (M = Y, Dy)[4] and $[MCl(OH_2)_2(18\text{-crown-6})]Cl_2.2H_2O$ (M = Sm, Gd, Tb)[5]. In $[Y_{(3)}(NO_3)_5]^{2-}$, the ten-coordinate Y^{3+} ion is coordinated to five bidentate nitrato groups. The bond lengths lie in the range Y(3)-O(NO_3^-), 2.388–2.451Å. The coordination geometry is a distorted bicapped square antiprism[3] (Figure 4), similar to those observed in $[(C_6H_5)_4As]_2[Eu(NO_3)_5]$[6] and $(NO)_2[Ho(NO_3)_5]$.[7]

It can be seen from Table 5 that some O(water)...O(crown ether) distances range from 2.819 to 2.982Å; these values lie in the range of hydrogen bonding distances.[8] This suggests that hydrogen bonding between coordinated water molecules and some

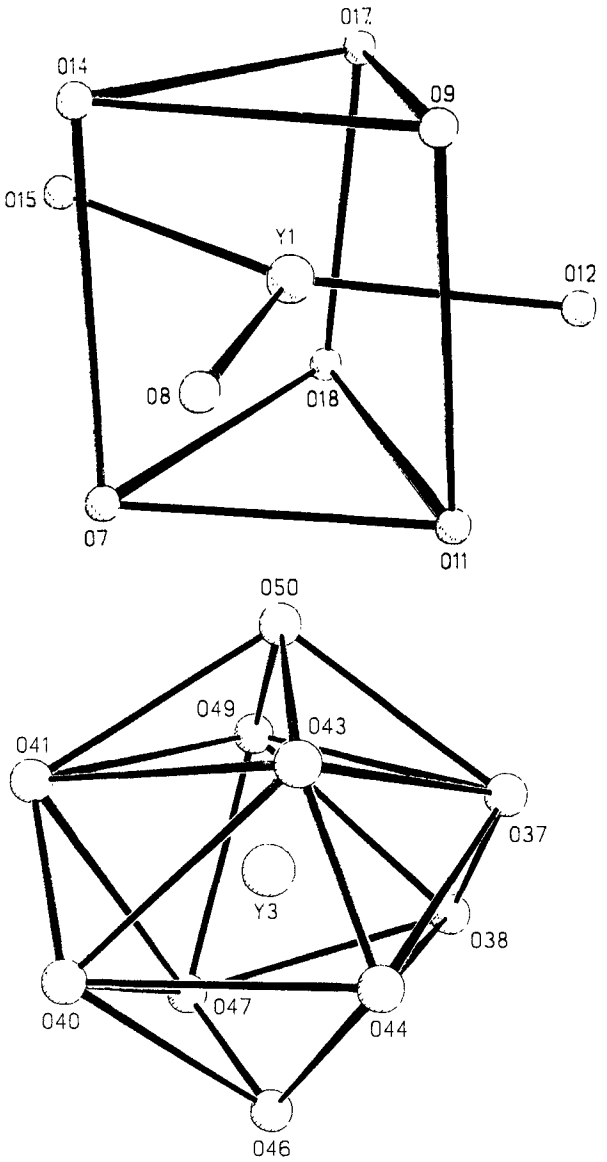

Figure 3 Coordination geometries.

Table 5 O(water)...O(crown ether) distances (Å)

Atoms	Distance	Atoms	Distance
O(17)...O(3)	2.844	O(17)...O(10)	2.819
O(18)...O(4)	2.862	O(18)...O(6)	2.865
O(35)...O(19)	2.912	O(35)...O(26)	2.982
O(36)...O(23)	2.824	O(36)...O(25)	2.891

of the crown ether oxygen atoms. This may be the reason that these water molecules are not replaced by crown ether oxygen atoms.

This study shows that when a small rare earth ion (Y^{3+}) reacts with a large cavity crown ether, all of the ether oxygen atoms in the crown ether cannot simultaneously coordinate to the metal ion. In order to achieve correct M-O(crown) distances, DB30C10 can only bond to Y^{3+} ion through some of ether oxygen atoms. To fulfil coordination number requirements of the Y^{3+} ion, only one of three nitrate groups is replaced by the crown ether, resulting in a singlely charged cation $[Y(NO_3)_2(OH_2)_2(DB30C10)]^+$, while two other nitrate groups coordinate to yttrium nitrate to form a doubly charged anion. This is the reason why the complex contains two cations and one anion and a metal to crown ether ratio of 3:2.

Acknowledgements

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Supplementary Material

Full lists of anisotropic thermal parameters, hydrogen atom positions, bond lengths and angles, and tables of observed and calculated structure factors are available from X.G.

References

1. J.-C.G. Bünzli, "Handbook on the Physics and Chemistry of Rare Earths", K.A. Gschneidner, Jr. and L. Eyring, Eds., (North Holland Publ. Co., Amsterdam, 1987), Vol. 9, p. 362.
2. M. Ciampolini and N. Nardi, *Inorg. Chim. Acta*, **32**, L9 (1979).
3. M.G.B. Drew, *Coord. Chem. Rev.*, **24**, 179 (1977).
4. R.D. Rogers, L.K. Kurihara and E.J. Voss, *Inorg. Chem.*, **26**, 2360 (1987).
5. R.D. Rogers and L.K. Kurihara, *Inorg. Chem.*, **26**, 1498 (1987).
6. J.-C.G. Bünzli, B. Klein, G. Chapuis and K.J. Schenk, *J. Inorg. Nucl. Chem.*, **42**, 1307 (1980).
7. G.E. Togood and C. Chieh, *Can. J. Chem.*, **53**, 831 (1975).
8. R.D. Rogers and E.J. Voss, *J. Coord. Chem.*, **16**, 405 (1988).