Lorentz and polarization corrections were applied to the intensity data. The structure was solved by standard Patterson methods and subsequently completed by Fourier syntheses. All non-H atoms were refined anisotropically. The H atoms of the water molecules were located on a ΔF map and refined with constraints, while the H atoms of bpm were set in calculated positions and refined as riding atoms. A common fixed isotropic parameter was assigned to all H atoms. Solution and refinement were performed with the *SHELXTL-Plus* system (Sheldrick, 1990). The final geometrical calculations were carried out with the *PARST* program (Nardelli, 1983). The graphical manipulations were performed using the *XP* utility of the *SHELXTL-Plus* system.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71836 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: NA1055]

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Yttrium(III) and Lanthanum(III) Complexes of a 15-Membered Macrocycle with Three Pendant Carboxymethyl Groups

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Abstract

The yttrium(III) and lanthanum(III) complexes of 9,14-dioxo-1,4,7,10,13-pentaaza-1,4,7-cyclopentadecanetriacetic acid, namely, diaquabis(9,14-dioxo-O^{14}:O^{9}-1,4,7,10,13-pentaaza-N^{1},N^{4}:N^{7}-1,4,7-cyclopentadecanetriacetato-O^{1},O^{4}:O^{7})diyttrium(III) tetradecahydrate $[Y_2(C_{16}H_{24}N_5O_8)_2(H_2O)_2].14H_2O$, and diaquabis(9,14-dioxo-O^{14}:O^{9}-1,4,7,10,13-pentaaza-N^{1},N^{4}:N^{7}-1,4,7-cyclopentadecanetriacetato-O^{1},O^{4}:-O^{7})dilanthanum(III) tetradecahydrate, $[La_2(C_{16}H_{24}N_5O_8)_2(H_2O)_2].14H_2O$, have a binuclear structure in which two metal ions are located between two macrocycles. The coordination geometry around each metal ion is distorted tricapped trigonal prismatic. The geometrical properties are defined by the inflexible conformation of the macrocycle and the strong coordination of the O atoms.

Comment

The 15-membered macrocycle with three pendant carboxymethyl groups 9,14-dioxo-1,4,7,10,13-pentaaza-1,4,7-cyclopentadecanetriacetic acid, abbreviated as (15-dten)H_3, forms a novel binuclear gadolinium(III) complex, $[Gd_2(15\text{-dten})_2(H_2O)_2]$, in which two Gd^{III} ions are located between two macrocycles (Inoue, Inoue, Muñoz, Bruck & Fernando, 1993). This finding prompted us to study the structures of the complexes (I) of other rare earth elements with a

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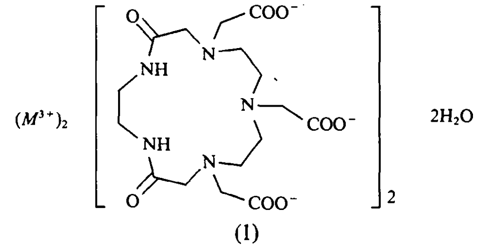
different ion size. In this paper, we report the structures of the La^{III} and the Y^{III} chelates of (15-dten)$^{3-}$; the former has the largest ionic radius among the rare earth elements and the ionic radius of the latter is significantly smaller than that of Gd^{III} (Lide, 1991).



(1)

Both metal complexes are isostructural with the corresponding Gd^{III} complex, and have a binuclear centrosymmetric molecular structure, as illustrated in Fig. 1, which represents the structure of the La^{III} complex. Some geometrical parameters of the Y^{III} and La^{III} complexes are collected in Table 2 together with the corresponding parameters for the Gd^{III} complex for comparison. Each metal ion in each complex is coordinated to nine atoms: O(1), O(3), O(5), N(1) and N(2) from one ligand molecule, O(2′), O(7′) and N(3′) from the second ligand molecule, and one water O atom, O(*w*1). The resulting coordination geometry is described as distorted tricapped trigonal prismatic, as shown in Fig. 2. The dihedral angles between the triangular planes are shown in Table 2, the distortion from the ideal D_{3h} symmetry being largest in the La^{III} complex.

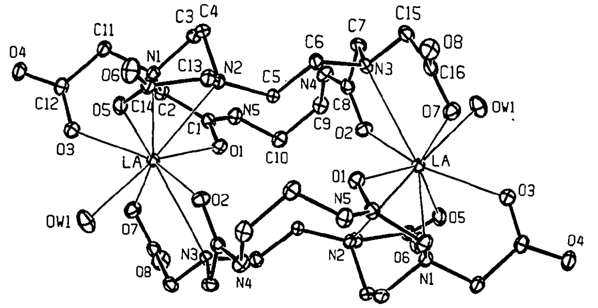


Fig. 1. Structure of $[La_2(15\text{-dten})_2(H_2O)_2]$. The atoms are shown at the 20% probability level. The structure of the Y^{III} compound is essentially identical.

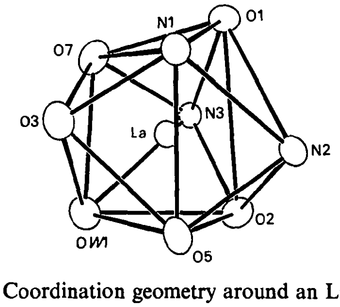


Fig. 2. Coordination geometry around an La^{III} ion.

The five atoms of each amide group —C—CO—N—C— are in a single plane (Table 3), resulting in the inflexibility of the macrocycle. The *M*—O distances are significnatly shorter that the *M*—N distances in every compound. The corresponding *M*—O distances of the three complexes differ as expected from the differences in ionic radii of the central metals. In contrast, the differences in *M*—N distances are smaller than those expected from the differences in the M^{3+} radii. The positions of the N atoms on the coordination sphere are defined by the strong coordination of the O atoms and the conformation of the macrocyclic ring.

Experimental

The compounds were prepared by essentially the same method as reported for the corresponding Gd complex (Inoue *et al.*, 1993). Crystals were obtained from an aqueous solution by slow evaporation. A crystal was sealed in a glass capillary together with the mother liquor.

$[Y_2(15\text{-dten})_2(H_2O)_2]$

Crystal data

$[Y_2(C_{16}H_{24}N_5O_8)_2(H_2O)_2]$.-
$14H_2O$
$M_r = 1294.86$
Orthorhombic
Pbca
$a = 18.316$ (2) Å
$b = 19.075$ (2) Å
$c = 15.607$ (2) Å
$V = 5453$ (2) Å^3
$Z = 4$
$D_x = 1.58$ Mg m^{-3}

Mo *Kα* radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10\text{-}15°$
$\mu = 2.23$ mm^{-1}
$T = 294$ (1) K
Plate
0.45 × 0.30 × 0.20 mm
Colorless

Data collection

Syntex Nicolet $P2_1$ diffractometer
θ-2θ scans
Absorption correction: empirical (ψ scan)
$T_{min} = 0.800$, $T_{max} = 1.000$
5343 measured reflections
4800 independent reflections

2108 observed reflections $[I > 3.0\sigma(I)]$
$\theta_{max} = 25°$
$h = 0 \rightarrow 21$
$k = 0 \rightarrow 22$
$l = 0 \rightarrow 18$
3 standard reflections monitored every 97 reflections intensity variation: none

Refinement

Refinement on *F*
$R = 0.047$
$wR = 0.065$
$S = 1.75$
2108 reflections
343 parameters
H-atom parameters not refined, included as riding atoms

$w = 4F_o^2/\sigma^2(F_o^2)$
$(\Delta/\sigma)_{max} = 0.09$
$\Delta\rho_{max} = 1.41$ (9) e Å^{-3}
$\Delta\rho_{min} = -0.42$ (9) e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

$[La_2(15\text{-dten})_2(H_2O)_2]$

Crystal data

$[La_2(C_{16}H_{24}N_5O_8)_2(H_2O)_2]$.-14$H_2O$
M_r = 1394.86
Orthorhombic
Pbca
a = 18.259 (1) Å
b = 19.181 (1) Å
c = 15.738 (1) Å
V = 5511.9 (6) Å^3
Z = 4
D_x = 1.68 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 6–10°
μ = 1.63 mm^{-1}
T = 292 (1) K
Plate
0.33 × 0.33 × 0.17 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω-2θ scans
Absorption correction: empirical (ψ scan)
T_{min} = 0.852, T_{max} = 1.000
5385 measured reflections
4840 independent reflections

3790 observed reflections [$I > 3.0\sigma(I)$]
R_{int} = 0.007
θ_{max} = 25°
$h = 0 \rightarrow 21$
$k = 0 \rightarrow 22$
$l = 0 \rightarrow 18$
3 standard reflections
frequency: 60 min
intensity variation: −3.4%

Refinement

Refinement on F
R = 0.022
wR = 0.034
S = 1.32
3790 reflections
343 parameters
H-atom parameters not refined, included as riding atoms

$w = 4F_o^2/\sigma^2(F_o^2)$
$(\Delta/\sigma)_{max}$ = 0.01
$\Delta\rho_{max}$ = 0.39 (6) e Å^{-3}
$\Delta\rho_{min}$ = −0.11 (6) e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$B_{eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
$[Y_2(15\text{-dten})_2(H_2O)_2]$				
Y	0.42741 (4)	0.45211 (5)	0.30519 (5)	2.39 (1)
O(1)	0.5519 (3)	0.4713 (3)	0.3479 (3)	2.6 (1)
O(2)	0.6356 (3)	0.5540 (3)	0.5624 (3)	2.6 (1)
O(3)	0.4106 (3)	0.4444 (3)	0.1568 (4)	3.2 (1)
O(4)	0.4008 (4)	0.4932 (4)	0.0270 (4)	3.6 (1)
O(5)	0.3245 (3)	0.5200 (3)	0.2800 (4)	3.3 (1)
O(6)	0.2315 (3)	0.5857 (4)	0.3289 (4)	4.2 (2)
O(7)	0.5000 (3)	0.6395 (3)	0.7481 (4)	3.2 (1)
O(8)	0.4116 (3)	0.7173 (3)	0.7224 (4)	3.8 (2)
N(1)	0.4975 (4)	0.5459 (4)	0.2179 (4)	2.5 (1)
N(2)	0.4237 (4)	0.5836 (4)	0.3786 (4)	2.4 (1)
N(3)	0.5314 (4)	0.6564 (4)	0.5715 (4)	2.3 (2)
N(4)	0.6828 (4)	0.5980 (4)	0.4396 (4)	2.9 (2)
N(5)	0.6684 (4)	0.4932 (4)	0.3009 (4)	2.9 (2)
C(1)	0.5960 (5)	0.4896 (5)	0.2910 (5)	2.7 (2)
C(2)	0.5679 (5)	0.5105 (5)	0.2032 (5)	3.0 (2)
C(3)	0.5110 (5)	0.6128 (5)	0.2635 (5)	3.1 (2)
C(4)	0.4451 (4)	0.6363 (4)	0.3137 (5)	2.7 (2)
C(5)	0.4715 (4)	0.5831 (4)	0.4560 (5)	2.4 (2)
C(6)	0.4668 (5)	0.6486 (5)	0.5141 (5)	2.8 (2)
C(7)	0.5973 (5)	0.6692 (5)	0.5210 (6)	2.9 (2)
C(8)	0.6400 (4)	0.6010 (5)	0.5080 (5)	2.2 (2)
C(9)	0.7358 (4)	0.5432 (5)	0.4263 (6)	3.3 (2)
C(10)	0.7035 (5)	0.4761 (5)	0.3829 (6)	3.2 (2)
C(11)	0.4601 (5)	0.5589 (5)	0.1363 (5)	3.4 (2)
C(12)	0.4211 (5)	0.4934 (5)	0.1041 (5)	2.9 (2)
C(13)	0.3462 (4)	0.5970 (5)	0.3992 (5)	2.8 (2)
C(14)	0.2986 (5)	0.5660 (5)	0.3314 (5)	2.9 (2)
C(15)	0.5162 (5)	0.7162 (5)	0.6297 (5)	2.9 (2)
C(16)	0.4717 (5)	0.6889 (5)	0.7051 (5)	3.0 (2)
O(w1)	0.3383 (3)	0.3642 (3)	0.2827 (4)	3.5 (1)
O(w2)	0.1211 (3)	0.3681 (4)	0.4741 (4)	4.6 (2)
O(w3)	0.4871 (4)	0.3155 (4)	0.0772 (5)	5.3 (2)
O(w4)	0.3588 (5)	0.1361 (5)	0.4749 (5)	7.9 (2)
O(w5)	0.1785 (4)	0.2123 (5)	0.1126 (6)	7.9 (3)
O(w6)	0.2946 (4)	0.3090 (5)	0.1217 (5)	7.5 (2)
O(w7)	0.2002 (4)	0.3303 (6)	0.3342 (5)	8.3 (3)
O(w8)	0.3398 (6)	0.1888 (6)	0.2084 (7)	11.9 (3)
$[La_2(15\text{-dten})_2(H_2O)_2]$				
La	0.42306 (1)	0.45096 (1)	0.30910 (1)	2.085 (3)
O(1)	0.5577 (1)	0.4719 (1)	0.3462 (1)	2.67 (4)
O(2)	0.6437 (1)	0.5554 (1)	0.5519 (1)	2.92 (4)
O(3)	0.4103 (1)	0.4485 (1)	0.1530 (2)	3.35 (5)
O(4)	0.4055 (1)	0.4984 (1)	0.0263 (1)	3.49 (5)
O(5)	0.3166 (1)	0.5295 (1)	0.2862 (1)	3.28 (4)
O(6)	0.2300 (1)	0.5971 (1)	0.3399 (2)	4.30 (5)
O(7)	0.5027 (1)	0.6478 (1)	0.7451 (1)	3.30 (4)
O(8)	0.4172 (1)	0.7257 (1)	0.7127 (2)	3.97 (5)
N(1)	0.4982 (1)	0.5484 (1)	0.2189 (2)	2.49 (5)
N(2)	0.4236 (1)	0.5866 (1)	0.3817 (1)	2.30 (5)
N(3)	0.5373 (1)	0.6586 (1)	0.5673 (1)	2.26 (4)
N(4)	0.6874 (1)	0.6014 (1)	0.4315 (2)	2.99 (5)
N(5)	0.6711 (1)	0.4968 (1)	0.2975 (2)	2.91 (5)
C(1)	0.6002 (2)	0.4914 (2)	0.2884 (2)	2.52 (6)
C(2)	0.5687 (2)	0.5124 (2)	0.2033 (2)	2.90 (6)
C(3)	0.5120 (2)	0.6149 (2)	0.2654 (2)	2.69 (6)
C(4)	0.4459 (2)	0.6381 (2)	0.3162 (2)	2.65 (6)
C(5)	0.4725 (2)	0.5857 (1)	0.4569 (2)	2.34 (5)
C(6)	0.4706 (2)	0.6508 (2)	0.5131 (2)	2.60 (6)
C(7)	0.6029 (2)	0.6705 (2)	0.5146 (2)	2.69 (6)
C(8)	0.6457 (2)	0.6038 (2)	0.4994 (2)	2.53 (6)
C(9)	0.7410 (2)	0.5464 (2)	0.4164 (2)	3.23 (6)
C(10)	0.7090 (2)	0.4816 (2)	0.3757 (2)	3.12 (6)
C(11)	0.4612 (2)	0.5621 (2)	0.1374 (2)	2.96 (6)
C(12)	0.4234 (2)	0.4978 (2)	0.1022 (2)	2.79 (6)
C(13)	0.3472 (2)	0.6012 (2)	0.4045 (2)	2.70 (6)
C(14)	0.2940 (2)	0.5741 (2)	0.3385 (2)	2.82 (6)
C(15)	0.5237 (2)	0.7187 (2)	0.6244 (2)	2.81 (6)
C(16)	0.4764 (2)	0.6964 (2)	0.6991 (2)	2.84 (6)
O(w1)	0.3244 (1)	0.3614 (1)	0.2791 (2)	4.34 (5)
O(w2)	0.1144 (2)	0.3651 (1)	0.4700 (2)	4.99 (6)
O(w3)	0.4829 (1)	0.3113 (1)	0.0809 (2)	4.68 (6)
O(w4)	0.3573 (2)	0.1321 (1)	0.4812 (2)	5.59 (7)
O(w5)	0.1770 (2)	0.2155 (2)	0.1016 (2)	8.17 (9)
O(w6)	0.2916 (2)	0.3069 (2)	0.1190 (2)	7.38 (9)
O(w7)	0.1906 (2)	0.3237 (3)	0.3277 (2)	10.6 (1)
O(w8)	0.3334 (2)	0.1901 (2)	0.2152 (2)	7.60 (9)

Table 2. *Comparison of geometric parameters* (Å, °) *for* $[M_2(15\text{-dten})_2(H_2O)_2].14H_2O$, *where* M = Y, La *or* Gd, *and dihedral angles* (°) *between planes in the tetradecahedron*

	Y	Gd	La
Metal-ion coordination			
M—O(1)$_{amide}$	2.403 (5) [−0.046]	2.449 (2)	2.559 (2) [+0.110]
M—O(2)$_{amide}$	2.370 (5) [−0.036]	2.406 (2)	2.507 (2) [+0.101]
M—O(3)$_{carboxylato}$	2.342 (5) [−0.038]	2.380 (3)	2.468 (2) [+0.088]
M—O(5)$_{carboxylato}$	2.320 (6) [−0.061]	2.381 (3)	2.484 (2) [+0.103]
M—O(7)$_{carboxylato}$	2.348 (6) [−0.033]	2.381 (3)	2.481 (2) [+0.100]
M—N(1)$_{amine}$	2.589 (7) [−0.022]	2.611 (3)	2.718 (2) [+0.107]
M—N(2)$_{amine}$	2.759 (7) [−0.002]	2.761 (3)	2.842 (2) [+0.081]
M—N(3)$_{amine}$	2.926 (7) [+ 0.011]	2.915 (3)	2.953 (2) [+0.038]
M—O(w1)$_{water}$	2.367 (6) [−0.045]	2.412 (3)	2.534 (2) [+0.122]
Ionic radius*	1.015 [−0.045]	1.06	1.18 [+0.13]

Dihedral angles			
O(1)—N(1)—O(7′) / O(3)—N(2)—N(3′)	21.4	22.4	25.3 (1)
O(2′)—O(5)—O(*w*1) / O(3)—N(2)—N(3′)	5.3	4.9	6.5 (2)

* Ionic radii of the trivalent ions with eight coordination bonds from Lide (1991).

Table 3. *Selected distances* (Å) *and torsion angles* (°) *for* $[M_2(15\text{-dten})_2(H_2O)_2].14H_2O$, *where* M = Y *or* La

	Y	La
C(1)—O(1)	1.25 (1)	1.253 (4)
C(8)—O(2)	1.24 (1)	1.243 (4)
C(12)—O(3)	1.26 (1)	1.262 (4)
C(12)—O(4)	1.26 (1)	1.239 (4)
C(14)—O(5)	1.28 (1)	1.257 (4)
C(14)—O(6)	1.29 (1)	1.249 (4)
C(16)—O(7)	1.27 (1)	1.274 (4)
C(16)—O(8)	1.26 (1)	1.238 (4)
C(1)—N(5)	1.34 (1)	1.307 (4)
C(8)—N(4)	1.33 (1)	1.313 (4)
C(9)—N(4)—C(8)—O(2)	−7.9 (13)	−7.4 (5)
C(9)—N(4)—C(8)—C(7)	168.7 (7)	168.9 (3)
C(10)—N(5)—C(1)—O(1)	−0.6 (14)	−0.7 (5)
C(10)—N(5)—C(1)—C(2)	178.7 (8)	177.4 (3)

The H atoms bonded to the C or N atoms were placed at idealized positions with bond lengths of 0.95 Å. Most H atoms of the water molecules were located from the ΔF map, but some H atoms, *i.e.* those associated with the O(*w*5), O(*w*6), O(*w*7) and O(*w*8) atoms in the Y complex, and the O(*w*8) atom in the La complex, could not be located. The located H atoms were included in the structure-factor calculations as riding atoms with fixed isotropic temperature factors ($B = 5\ \text{Å}^2$). For $[Y_2(15\text{-dten})_2(H_2O)_2]$, the *UCLA Crystallographic Package* (1984) was used for data collection and cell refinement; *SDP* (Frenz, 1978) was used to solve and refine the structure. For $[La_2(15\text{-dten})_2(H_2O)_2]$, *MolEN* (Fair, 1990) was used to solve and refine the structure and the diagrams were prepared using *ORTEP*II (Johnson, 1976).

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Lists of experimental details, structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, bond distances and angles involving H atoms, hydrogen-bonding geometry, least-squares-planes data and torsion angles, along with packing diagrams for both compounds, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71741 (53 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BR1049]

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(Bipyridyl-*N*,*N*′)diiodoplatinum(II)

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Abstract

Square-planar (bipyridyl-*N*,*N*′)diiodoplatinum(II), $[PtI_2(C_{10}H_8N_2)]$, has normal Pt—N(bipyridyl) [2.029 (7) Å] and Pt—I bonds [2.589 (2) Å]. The bipyridyl ligand exhibits normal distances and angles. Because of steric effects, the intramolecular I···I separation [3.587 (1) Å] and the corresponding I—Pt—I angle [87.7 (1)°] are significantly smaller than those observed for other *cis*-bis(iodo)bis(*N*-donor) complexes. The parallel square-planar units stack to form a chain structure. Relative lateral displacement of consecutive molecules along a chain results in a Pt···Pt distance [5.291 (1) Å] considerably longer than the interplanar spacing [3.510 (11) Å].

Comment

In our work on the spectroscopy of linear-chain platinum(II) diimines, we have examined a series of $Pt(bpy)X_2$ complexes (X = Cl, Br, I). The bis(chloro) complex exhibits dimorphism, crystallizing in a yellow form as discrete monomer units (Textor & Oswald, 1974) and in a red form as a linear chain with a Pt···Pt separation of 3.45 Å (Textor & Oswald, 1974; Osborn & Rogers, 1974). The spectroscopic properties of the yellow bis(bromo) analogue

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