Cl1—Ge—Cl3	95.51 (3)	Cl3^{i}—Ge—Cl1^{ii}	106.920 (16)
Cl1—Ge—Cl2	95.34 (3)	Cl1—Ge—Cl2^{iii}	107.96 (3)
Cl3—Ge—Cl2	95.94 (3)	Cl3—Ge—Cl2^{iii}	84.30 (2)
Cl1—Ge—Cl3^{i}	78.86 (2)	Cl2—Ge—Cl2^{iii}	156.59 (4)
Cl3—Ge—Cl3^{i}	171.536 (16)	Cl3^{i}—Ge—Cl2^{iii}	91.391 (16)
Cl2—Ge—Cl3^{i}	90.91 (3)	Cl1^{ii}—Ge—Cl2^{iii}	73.983 (17)
Cl1—Ge—Cl1^{ii}	173.99 (3)	C1—P—C2	111.2 (2)
Cl3—Ge—Cl1^{ii}	78.93 (2)	C1—P—C3	110.91 (19)
Cl2—Ge—Cl1^{ii}	83.07 (3)	C2—P—C3	109.55 (19)

Symmetry codes: (i) $\frac{1}{2} + x, \frac{1}{2} - y, 2 - z$; (ii) $x, \frac{1}{2} - y, \frac{1}{2} - z$; (iii) $\frac{1}{2} - x, \frac{1}{2} - y, z$.

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
P—H···Cl3^{i}	1.28 (3)	2.84 (3)	3.6865 (11)	146.3 (5)
C1—H1*C*···Cl3^{ii}	0.92 (5)	2.83 (5)	3.649 (4)	149 (4)
C3—H3*A*···Cl3^{iii}	0.96 (5)	2.81 (5)	3.725 (4)	159 (3)
C3—H3*C*···Cl1^{iv}	0.93 (5)	2.84 (5)	3.647 (4)	146 (4)

Symmetry codes: (i) $\frac{1}{2} - x, y, \frac{3}{2} - z$; (ii) $\frac{1}{2} - x, \frac{1}{2} - y, z$; (iii) $\frac{1}{2} + x, \frac{1}{2} - y, 2 - z$; (iv) $x, \frac{1}{2} - y, \frac{1}{2} - z$.

The structure was solved by the Patterson method. All non-H atoms were refined anisotropically and all H atoms were refined isotropically. The program *XSHAPE* (Stoe, 1997) was used for crystal optimization before a numerical absorption correction implemented in *X-RED* (Stoe & Cie, 1996) was applied. 254 strong reflections with more than six symmetry equivalents for each reflection were used for the optimization. *XSHAPE* is based on the program *HABITUS* (Herrendorf, 1993).

Data collection: *IPDS Software* (Stoe, 1998); cell refinement: *CELL* in *IPDS Software*; data reduction: *INTEGRATE* in *IPDS Software*; program used to prepare the *SHELX* input files: *UTILITY* (Pickardt, 1994); program used to solve and refine structure: *SHELX*97 (Sheldrick, 1997); molecular graphics: *DIAMOND* (Bergerhoff, 1996) and *PLUTON*92 (Spek, 1992); software used to prepare material for publication: *SHELX*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: JZ1290). Services for accessing these data are described at the back of the journal.

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A holmium complex of a macrocyclic ligand (DOTA) and its isostructural europium analogue

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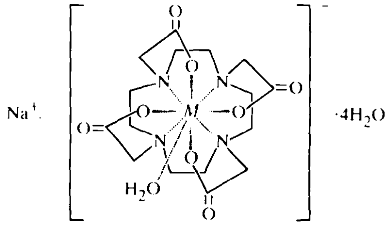
Abstract

The holmium and europium complexes of the tetraanion of 1,4,7,10-tetraazacyclododecane-1,4,7,10-tetraacetic acid (H_4DOTA), namely, sodium aqua(1,4,7,10-tetraazacyclododecane-1,4,7,10-tetraacetato)holmium(III) tetrahydrate, $Na[Ho(C_{16}H_{24}N_4O_8)(H_2O)]\cdot 4H_2O$, and sodium aqua(1,4,7,10-tetraazacyclododecane-1,4,7,10-tetraacetato)europium(III) tetrahydrate, $Na[Eu(C_{16}H_{24}N_4O_8)(H_2O)]\cdot 4H_2O$, show nine-coordination around the metal ion, with a square antiprismatic coordination geometry involving four N- and four O-donor atoms of the DOTA ligand, and a coordinated water molecule in a capping position.

Comment

Lanthanide(III) complexes with polydentate ligands are of interest because of the possibility of obtaining, with the encapsulation of the metal, stable derivatives which are useful as contrast agents in magnetic resonance imaging (Lauffer, 1987, 1990). The high thermodynamic stability and kinetic inertness of complexes with the macrocyclic ligand DOTA, essential for their *in vivo* use (Desreux, 1980; Wang *et al.*, 1992; Tóth *et al.*, 1994), suggested to us a systematic investigation of their structural features. Numerous studies dealing with their solution behaviour and different isomeric forms, differing in the arrangement of the acetate arms, have been postulated on the basis of their NMR spectra (Aime *et al.*, 1992; Howard *et al.*, 1998). The reported X-ray crystal structures of the Ln^{III}–DOTA complexes Na[Ln(DOTA)(H_2O)]·$4H_2O$ show that the ligand provides eight donor sites (four O and four N atoms) in a square antiprismatic geometry, with a water molecule capping the O_4 plane, for Eu (Spirlet *et al.*, 1984), Gd (Dubost *et al.*, 1991; Chang *et al.*, 1993) and Lu (Aime *et al.*, 1996), while for La, an unexpected polymeric derivative, Na[La(HDOTA)La(DOTA)]·$10H_2O$ (Aime, Barge *et al.*, 1997), has been found due to the coordination in the capping position by the oxygen of a carboxyl group of the adjacent unit instead of a water molecule. Another feature is the different orientation of the O_4 plane with respect to the N_4 plane, with a twist angle of about 39° for the structures of the Eu, Gd, Lu and Y complexes (Parker *et al.*, 1994), but of 21 and 23° for the two independent La coordination spheres, defined as 'inverted square antiprism' to differentiate them from the square antiprism found in the other elements of the series (Aime, Botta *et al.*, 1997). We have now investigated the structures of the Ho derivative, (I), and of a second Eu derivative (form *B*), (II), which are isomorphous.



(I) *M* = Ho
(II) *M* = Eu

The unit-cell dimensions of form *B* differ from those of the compound reported previously (form *A*) (Spirlet *et al.*, 1984) mainly in the angle α [82.83 (3) *versus* 92.87 (1)°]; it seemed possible that we had a different isomer but the crystal structure shows that it is a different polymorph of the same isomer. The Ho and Eu (form *B*) complexes are isomorphous with the analogous Y, Gd and Lu derivatives, with the usual monocapped square antiprismatic coordination geometry. The additional water molecules form a hydrogen-bond network with the coordinated water molecule and the O atoms of the carboxyl groups. The twist angle of the two squares formed respectively by the coordinated O and N atoms is again about 39°. The conformation and atom labelling for the holmium derivative is shown in Fig. 1. The average Ho—N and Eu—N bond distances of 2.641 (2) and 2.677 (2) Å, respectively, in the current structures, are larger than the average Ho—O [2.330 (2) Å] and Eu—O [2.379 (2) Å] distances found in the other members of the isomorphous series. In form *A* (Spirlet *et al.*, 1984), the averaged Eu—N [2.680 (3) Å] and Eu—O [2.393 (3) Å] bond lengths are not significantly different from those in form *B*, but the range of bond lengths is much greater [Eu—N: 2.519 (3)–2.900 (3) Å in form *A* and 2.660 (2)–2.704 (2) Å in form *B*; Eu—$O_{carboxylic}$: 2.247 (3)–2.511 (3) Å in form *A* and 2.371 (2)–2.390 (2) Å in form *B*].

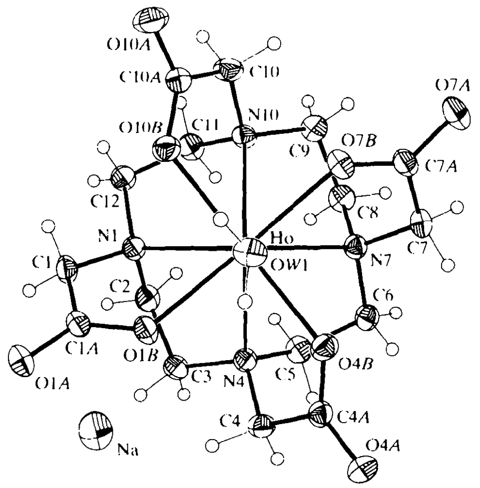


Fig. 1. The cation and anion in Na[Ho(DOTA)(H_2O)]·$4H_2O$. Displacement ellipsoids are drawn at the 40% probability level. The non-coordinated water molecules are not shown.

Experimental

The title Ho and Eu complexes were prepared as described elsewhere (Desreux, 1980). Crystals were obtained by diffusion methods from a water and acetone solution (room temperature) after several days.

Compound (I)

Crystal data

$Na[Ho(C_{16}H_{24}N_4O_8)(H_2O)]\cdot 4H_2O$
M_r = 678.39
Triclinic
$P\bar{1}$
a = 8.735 (2) Å
b = 9.093 (2) Å
c = 15.640 (3) Å
α = 82.94 (3)°
β = 85.85 (2)°
γ = 81.76 (2)°
V = 1218.2 (5) Å^3
Z = 2
D_x = 1.849 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 30 reflections
θ = 12–15°
μ = 3.34 mm^{-1}
T = 295 (2) K
Prismatic
0.42 × 0.38 × 0.34 mm
Pale yellow

Data collection

Philips PW1100 four-circle diffractometer
Profile-fitted $\theta/2\theta$ scans (Lehmann & Larsen, 1974)
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.247$, $T_{\max} = 0.321$
5231 measured reflections
5033 independent reflections
5032 reflections with $I > 3\sigma(I)$
$R_{\rm int} = 0.013$
$\theta_{\max} = 28.01°$
$h = -11 \rightarrow 11$
$k = -11 \rightarrow 12$
$l = 0 \rightarrow 20$
3 standard reflections
frequency: 180 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 3\sigma(F^2)] = 0.024$
$wR(F^2) = 0.061$
$S = 1.069$
5032 reflections
445 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0486P)^2 + 0.5937P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.009$
$\Delta\rho_{\max} = 1.758$ e Å^{-3} (close to Ho)
$\Delta\rho_{\min} = -1.717$ e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.0077 (7)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °) for (I)*

Ho—O7*B*	2.323 (2)	Ho—N10	2.665 (2)
Ho—O4*B*	2.330 (2)	Na—O4$A^{\rm i}$	2.323 (3)
Ho—O10*B*	2.330 (2)	Na—O1*A*	2.384 (3)
Ho—O1*B*	2.336 (2)	Na—O10$A^{\rm ii}$	2.402 (3)
Ho—O*W*1	2.443 (2)	Na—O*W*2	2.452 (5)
Ho—N7	2.628 (2)	Na—O1*B*	2.467 (3)
Ho—N1	2.631 (2)	Na—O10$B^{\rm ii}$	2.554 (3)
Ho—N4	2.642 (2)		
O7*B*—Ho—O4*B*	86.75 (8)	O1*B*—Ho—O*W*1	72.86 (7)
O7*B*—Ho—O10*B*	81.41 (8)	N7—Ho—N1	104.99 (8)
O4*B*—Ho—O10*B*	142.57 (7)	N7—Ho—N4	68.42 (8)
O7*B*—Ho—O1*B*	144.59 (7)	N1—Ho—N4	68.85 (7)
O4*B*—Ho—O1*B*	83.78 (8)	N7—Ho—N10	67.90 (8)
O10*B*—Ho—O1*B*	85.68 (8)	N1—Ho—N10	67.85 (8)
O7*B*—Ho—O*W*1	71.88 (8)	N4—Ho—N10	105.04 (8)

Symmetry codes: (i) $1 - x, 1 - y, 1 - z$; (ii) $1 - x, -y, 1 - z$.

Compound (II)

Crystal data

$\rm Na[Eu(C_{16}H_{24}N_4O_8)(H_2O)]\cdot 4H_2O$
$M_r = 665.42$
Triclinic
$P\bar{1}$
$a = 8.734$ (2) Å
$b = 9.116$ (3) Å
$c = 15.773$ (3) Å
$\alpha = 82.83$ (3)°
$\beta = 85.34$ (3)°
$\gamma = 81.43$ (2)°
$V = 1229.6$ (5) Å^3
$Z = 2$
$D_x = 1.797$ Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 30 reflections
$\theta = 13$–$17°$
$\mu = 2.64$ mm^{-1}
$T = 293$ (2) K
Prismatic
0.44 × 0.26 × 0.20 mm
Colourless

Data collection

Philips PW1100 four-circle diffractometer
Profile fitted $\theta/2\theta$ scans (Lehmann & Larsen, 1974)
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.450$, $T_{\max} = 0.590$
4837 measured reflections
4652 independent reflections
4615 reflections with $I > 3\sigma(I)$
$R_{\rm int} = 0.042$
$\theta_{\max} = 26°$
$h = -10 \rightarrow 10$
$k = -11 \rightarrow 11$
$l = 0 \rightarrow 19$
3 standard reflections
frequency: 180 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 3\sigma(F^2)] = 0.019$
$wR(F^2) = 0.052$
$S = 1.109$
4615 reflections
437 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + 1.4054P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.041$
$\Delta\rho_{\max} = 0.862$ e Å^{-3}
$\Delta\rho_{\min} = -1.136$ e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.0155 (6)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 2. *Selected geometric parameters (Å, °) for (II)*

Eu—O7*B*	2.371 (2)	Eu—N10	2.704 (2)
Eu—O10*B*	2.373 (2)	Na—O4$A^{\rm i}$	2.332 (3)
Eu—O4*B*	2.380 (2)	Na—O1*A*	2.405 (3)
Eu—O1*B*	2.390 (2)	Na—O10$A^{\rm ii}$	2.433 (3)
Eu—O*W*1	2.483 (2)	Na—O*W*2	2.446 (4)
Eu—N7	2.660 (2)	Na—O1*B*	2.461 (2)
Eu—N1	2.664 (2)	Na—O10$B^{\rm ii}$	2.513 (2)
Eu—N4	2.677 (2)		
O7*B*—Eu—O10*B*	81.89 (7)	O1*B*—Eu—O*W*1	73.44 (7)
O7*B*—Eu—O4*B*	87.15 (7)	N7—Eu—N1	104.09 (7)
O10*B*—Eu—O4*B*	144.13 (7)	N7—Eu—N4	67.99 (7)
O7*B*—Eu—O1*B*	146.33 (7)	N1—Eu—N4	68.25 (7)
O10*B*—Eu—O1*B*	86.48 (7)	N7—Eu—N10	67.42 (7)
O4*B*—Eu—O1*B*	84.02 (7)	N1—Eu—N10	67.57 (7)
O7*B*—Eu—O*W*1	73.03 (7)	N4—Eu—N10	104.22 (7)

Symmetry codes: (i) $1 - x, 1 - y, 1 - z$; (ii) $1 - x, -y, 1 - z$.

Weak reflections were skipped after a pre-scan. The H atoms were located in difference maps (except for those of O*W*2 and O*W*5 of the Eu derivative, which were omitted from the refinement) and refined with individual isotropic displacement parameters.

For both compounds, data collection: *FEBO* (Belletti, 1993); cell refinement: *FEBO*; data reduction: *FEBO*; program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *PARST*94 (Nardelli, 1995).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1277). Services for accessing these data are described at the back of the journal.

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Lead(II) oxydiacetate hydrate

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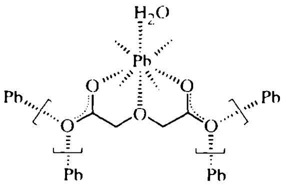
Abstract

The coordination polyhedron around the Pb atom in the title compound, poly[aqualead(II)-μ_5-oxydiacetato], $[Pb(\mu_5\text{-}C_4H_4O_5)(H_2O)]_n$, is a distorted square antiprism formed by three O atoms from one oxydiacetate ligand, four O atoms from four other identical ligands and one water O atom, resulting in a three-dimensional network structure. The almost planar oxydiacetate ligand is disordered over two positions. The Pb lone-pair $6s^2$ electrons show stereochemical activity in the coordination sphere.

Comment

The structure determination of the title compound is part of a study of interactions between *p*-block metals and small ligands of the type $[O_2C\text{–}CH_2\text{–}X\text{–}CH_2\text{–}CO_2]^{2-}$, where X can be NH, O or S. This type of ligand can be mono-, bi- or tridentate and may form bridges between the metal atoms. A variety of solid-state complexes with the oxydiacetate ion, $O(CH_2COO^-)_2$, have been investigated, *e.g.* complexes with the trivalent lanthanoids (Albertsson, 1970; Albertsson & Elding, 1976), Rb^+ (Albertsson *et al.*, 1973) and Sr^{2+} (Baggio *et al.*, 1996). In complexes with Pb^{2+}, the $6s^2$ lone pair is likely to affect the coordination geometry, resulting in irregular coordination polyhedra with a large range of Pb—O distances. Several Pb^{2+} complexes with active lone pairs are known, *e.g.* $[Pb(C_3H_2O_4)]$ (Bensch & Günter, 1987) and $[\{HB(3,5\text{-}Me_2pz)_3\}Pb(3,5\text{-}Me_2pzH)_3\text{-}Cl]$ (pz is pyrazolyl; Reger *et al.*, 1994).

Another reason for the present study was to compare the resulting structure with that of the related oxydiacetate double salt $[Pb_4(C_4H_4O_5)_3(NO_3)_2(H_2O)]$ (Svensson *et al.*, 1998), which involves three independent Pb atoms with coordination numbers seven, eight and nine. At least, the surroundings of the seven- and eight-coordinated Pb atoms are influenced by stereochemically active lone pairs. In order to eliminate nitrate from the structure, lead acetate was used as a precursor instead of lead nitrate, resulting in the polymeric compound $[\{Pb(H_2O)\}(\mu_5\text{-}C_4H_4O_5)]_n$, (I).



(I)

In the asymmetric unit of the title compound, the Pb atom, the water O atoms and the ether O1 atom of the oxydiacetate ligand are located on the $y = \frac{1}{4}$ mirror plane, with the acetate moieties distributed over two locations, *a* and *b*, with refined occupancies of 0.44 (3) and 0.56 (3), respectively (see *Experimental*). The differences between the *a* and *b* ligands are very small. They are almost planar, with r.m.s. deviations of 0.064 (for *a*) and 0.102 Å (for *b*), and have the expected overall geometry when compared with the