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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: OH1068). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Aquatris(dichloroacetato)ethanolbis(1,10-phenanthroline)lanthanum(III) and the Corresponding Samarium Complex

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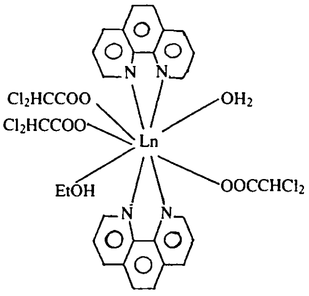
Abstract

The title complexes, [$La(CHCl_2CO_2)_3(C_{12}H_8N_2)_2$-($C_2H_5OH)(H_2O)$] and [$Sm(CHCl_2CO_2)_3(C_{12}H_8N_2)_2$-($C_2H_5OH)(H_2O)$], are isostructural. The La and Sm atoms are nine-coordinate with distorted tricapped trigonal prismatic coordination geometry. The average bond distances in the two structures are La—O(dichloroacetate) 2.470 (14), La—O(H_2O and C_2H_5OH) 2.59 (9), La—N 2.72 (2), Sm—O(dichloroacetate) 2.39 (3), Sm—O(H_2O and C_2H_5OH) 2.54 (13) and Sm—N 2.63 (4) Å. The complexes form one-dimensional intermolecular-hydrogen-bonded chains in a direction nearly parallel to the *a* axis. This stabilizes the crystal packing.

Comment

Rare earth dichloroacetates have been studied previously (Sing, Buhtra, Vaishnava & Misra, 1978; Spacu & Antonescu, 1969), particularly from the point of view of their preparation. However, a full understanding of the chemistry of these compounds is limited by the absence of structural information. Structural studies of lanthanide complexes involving carboxylate ligands have shown that the rare earth atoms have several types of coordination, usually with coordination numbers of eight or nine. In complexes containing the same ligands, the heavier rare earth atoms often coordinate to fewer ligands than the lighter ones. The carboxylate ligand has various modes of coordination to lanthanide atoms: monodentate, bidentate, bridging-bidentate or bridging-tridentate (Bone, Sowerby & Verma, 1978; Harrison, Giorgetti & Bunzle, 1985; Sawase, Koizumi, Suzuki, Shimoi & Ouchi, 1984). We have reported the synthesis and structure of dimeric $Gd(CCl_3COO)_3(phen)(EtOH)_2$ (phen = 1,10-phenanthroline) (Mao, Lu & Dong, 1989). As a part of our continuing study of dichloroacetate lanthanide complexes with a series of heterocyclic amine ligands, we have now synthesized and studied the complexes of lanthanide dichloroacetate with two 1,10-phenanthroline ligands (Ln from La to Yb), a series which includes more than one type of complex. The present paper describes the crystal structures of [$Ln(CHCl_2COO)_3(phen)_2(H_2O)(EtOH)$] [Ln = La (1) or Sm (2)].



(1) Ln = La
(2) Ln = Sm

The two complexes are isostructural; the structure of the La complex is shown in Fig. 1. The central atom is nine-coordinate: four N atoms from two phenanthroline ligands, three O atoms from monodentate dichloroacetate (dca) ions, one O atom from ethanol and one O atom from water form a distorted tricapped trigonal prism. The atoms O(3), O(5), O(7), N(2), N(3) and N(4) form the prism with O(1), O(8) and N(1) capping the rectangular faces. The average bond distances in the two structures are: La—O(dca) 2.470 (14), La—O(H_2O and EtOH) 2.59 (9), La—N 2.72 (2), Sm—O(dca) 2.39 (3), Sm—O(H_2O and EtOH) 2.54 (13) and Sm—N 2.63 (4) Å. A comparison of the metal–ligand bond distances in the two complexes shows that the bonds are shorter in (2) than in (1), which is to be expected from the lanthanide contraction. None of the atoms of the phenanthroline ligands deviates significantly from the least-squares plane through each ligand. The structures of the phen ligands are all identical and are similar to that of the phen ligand in $[Ln(CCl_3COO)_3(phen)(EtOH)]_2$ (Dong, Robertson, Wang & Barton, 1990), which possesses local C_{2v} symmetry. The dihedral angle between the least-squares planes of the two phen ligands is 28.93 (13) for (1) and 29.10 (14)° for (2). A Cl atom in one of the dichloroacetate ions is disordered, which suggests that the libration of the $CHCl_2$ group around the C—C bond is relatively large. A similar phenomenon was observed in the lanthanide trichloroacetate complex (Wang, Dong, Wu & Zheng, 1991).

As shown in Table 3, the ethanol and water molecules form hydrogen bonds with uncoordinated O atoms from the dca groups. Besides the intramolecular hydrogen bonds, one of the H atoms from H_2O is bonded to the O(6) atom in the next unit at $-x, y, z$. The entire structure contains one-dimensional chains of intermolecular hydrogen bonds of the type O—H···O in a direction nearly parallel to the a axis, which stabilizes the crystal packing.

The IR spectra of $[Ln(CHCl_2COO)_3(phen)_2(H_2O)(EtOH)]$ complexes (Ln = La, Ce, Pr, Nd and Sm) are all similar and the compositions of the complexes are in fair agreement with elemental analyses. The Ce, Pr and Nd complexes may, therefore, have the same structure as the La and Sm complexes.

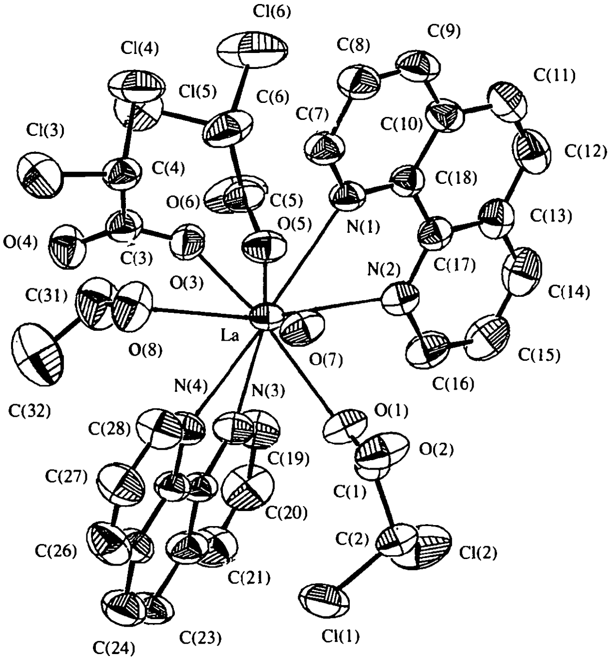


Fig. 1. The molecular structure and the numbering scheme for the lanthanum complex. Displacement ellipsoids are shown at the 50% probability level.

Experimental

The title complexes were prepared by similar methods. The relevant lanthanide oxide was dissolved completely in dichloroacetatic acid ($Ln:CHCl_2COOH$ 1:1.4 molar ratio) to obtain a solution of lanthanide dichloroacetate. After the solution had been evaporated slowly, the dry residue was recrystallized in water and the crystals were dried. 0.15 mmol of lanthanide dichloroacetate hydrate was dissolved completely in 12 ml of a mixed solvent of ethanol and water (1:1 *v*/*v*) and then mixed with a 0.3 mmol solution of phen in the same solvent. After a few days, single crystals suitable for X-ray work were obtained by slow evaporation. Analyses: calculated for (1), C 40.58, H 2.87, N 5.91, La 14.65%; found, C 40.37, H 2.65, N 5.93, La 14.66%; calculated for (2), C 40.10, H 2.84, N 5.85, Sm 15.68%; found, C 40.06, H 2.87, N 5.80, Sm 15.79%.

Complex (1)

Crystal data

$[La(CHCl_2CO_2)_3(C_{12}H_8N_2)_2(C_2H_5OH)(H_2O)]$
$M_r = 947.21$
Triclinic
$P\bar{1}$
$a = 8.554\,(1)$ Å
$b = 11.359\,(2)$ Å
$c = 19.748\,(6)$ Å
$\alpha = 76.43\,(2)°$
$\beta = 81.85\,(1)°$
$\gamma = 81.35\,(4)°$
$V = 1832.7\,(6)$ Å^3
$Z = 2$
$D_x = 1.716$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 11–15°$
$\mu = 1.66$ mm^{-1}
$T = 293$ K
Prism
0.45 × 0.20 × 0.20 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan
$T_{min} = 0.743$, $T_{max} = 1.000$
5919 measured reflections
5730 independent reflections
5204 observed reflections [$I > 3\sigma(I)$]

$R_{int} = 0.01$
$\theta_{max} = 24°$
$h = -9 \rightarrow 9$
$k = -13 \rightarrow 13$
$l = -22 \rightarrow 0$
3 standard reflections monitored every 200 reflections
intensity decay: 0.5%

Refinement

Refinement on F
$R = 0.044$
$wR = 0.047$
$S = 1.26$
5204 reflections
423 parameters
Unit weights applied

$(\Delta/\sigma)_{max} = 0.03$
$\Delta\rho_{max} = 2.57$ e Å^{-3}
$\Delta\rho_{min} = -1.12$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Complex (2)

Crystal data

$[Sm(CHCl_2CO_2)_3(C_{12}H_8N_2)_2(C_2H_5OH)(H_2O)]$

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å

M_r = 958.71
Triclinic
$P\bar{1}$
a = 8.476 (4) Å
b = 11.302 (2) Å
c = 19.631 (6) Å
α = 77.16 (2)°
β = 82.03 (3)°
γ = 81.36 (2)°
V = 1801.8 (9) Å^3
Z = 2
D_x = 1.767 Mg m^{-3}

Cell parameters from 25 reflections
θ = 11–15°
μ = 2.14 mm^{-1}
T = 293 K
Prism
0.35 × 0.15 × 0.15 mm
Light yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan
$T_{\min}$ = 0.820, $T_{\max}$ = 0.999
6338 measured reflections
6318 independent reflections
5404 observed reflections [$I > 3\sigma(I)$]

$R_{\rm int}$ = 0.021
$\theta_{\max}$ = 25°
$h = -10 \rightarrow 10$
$k = -13 \rightarrow 13$
$l = -23 \rightarrow 0$
3 standard reflections monitored every 200 reflections
intensity decay: 0.6%

Refinement

Refinement on F
R = 0.041
wR = 0.046
S = 1.87
5404 reflections
423 parameters
Unit weights applied

$(\Delta/\sigma)_{\max}$ = 0.63
$\Delta\rho_{\max}$ = 2.273 e Å^{-3}
$\Delta\rho_{\min}$ = −0.885 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) *for complexes* (1) *and* (2)

$$B_{\rm eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
Complex (1)				
La	0.13290 (4)	0.25134 (3)	0.26272 (2)	2.659 (6)
Cl(1)	0.0537 (3)	−0.0397 (2)	0.08782 (9)	5.95 (5)
Cl(2)	0.2736 (3)	−0.1643 (2)	0.1870 (1)	7.36 (5)
Cl(3)	−0.3284 (2)	0.7396 (2)	0.2294 (1)	6.05 (5)
Cl(4)	−0.1746 (3)	0.6475 (2)	0.3555 (1)	6.77 (5)
Cl(5)	0.3309 (5)	0.5836 (2)	0.3391 (2)	13.1 (1)
Cl(6)†	0.4500 (7)	0.4451 (5)	0.4518 (2)	9.7 (1)
Cl(6′)†	0.3137 (5)	0.3910 (3)	0.4625 (2)	5.32 (9)
O(1)	0.1099 (5)	0.0556 (4)	0.2313 (2)	4.08 (9)
O(2)	−0.1326 (5)	0.0042 (4)	0.2376 (3)	5.5 (1)
O(3)	−0.0429 (5)	0.4420 (4)	0.2692 (2)	4.1 (1)
O(4)	−0.0114 (5)	0.6153 (4)	0.1907 (2)	4.9 (1)
O(5)	0.3667 (5)	0.2859 (4)	0.3119 (2)	4.20 (9)
O(6)	0.5839 (5)	0.3490 (4)	0.3324 (3)	6.2 (1)
O(7)	−0.1569 (5)	0.2162 (4)	0.2780 (2)	3.97 (9)
O(8)	0.2523 (5)	0.4496 (4)	0.1858 (3)	5.2 (1)
N(1)	0.0337 (6)	0.2299 (4)	0.4032 (2)	3.2 (1)
N(2)	0.2489 (6)	0.0536 (4)	0.3586 (2)	3.5 (1)
N(3)	0.3584 (5)	0.1992 (4)	0.1596 (2)	3.5 (1)
N(4)	0.0542 (6)	0.2991 (5)	0.1306 (2)	3.6 (1)
C(1)	0.0116 (7)	−0.0057 (5)	0.2202 (3)	3.3 (1)
C(2)	0.0744 (7)	−0.1058 (5)	0.1782 (3)	3.9 (1)
C(3)	−0.0803 (7)	0.5522 (5)	0.2424 (3)	3.5 (1)
C(4)	−0.2315 (7)	0.6089 (5)	0.2812 (3)	3.8 (1)
C(5)	0.4403 (7)	0.3484 (5)	0.3366 (3)	3.6 (1)
C(6)	0.3285 (9)	0.4341 (6)	0.3782 (4)	5.7 (2)
C(7)	−0.0800 (8)	0.3108 (6)	0.4255 (3)	4.0 (1)
C(8)	−0.1203 (8)	0.3124 (6)	0.4969 (3)	4.6 (2)
C(9)	−0.0424 (8)	0.2292 (6)	0.5454 (3)	4.5 (1)
C(10)	0.0769 (7)	0.1406 (6)	0.5246 (3)	3.8 (1)
C(11)	0.1618 (8)	0.0486 (7)	0.5732 (3)	4.8 (2)
C(12)	0.2720 (8)	−0.0353 (7)	0.5512 (4)	4.8 (2)
C(13)	0.3068 (7)	−0.0358 (6)	0.4781 (3)	3.9 (1)
C(14)	0.4182 (8)	−0.1245 (6)	0.4539 (4)	5.1 (2)
C(15)	0.4412 (8)	−0.1226 (6)	0.3836 (4)	5.3 (2)
C(16)	0.3532 (8)	−0.0342 (6)	0.3382 (4)	4.4 (1)
C(17)	0.2260 (6)	0.0526 (5)	0.4288 (3)	3.2 (1)
C(18)	0.1096 (6)	0.1448 (5)	0.4518 (3)	3.1 (1)
C(19)	0.5042 (8)	0.1508 (6)	0.1720 (4)	4.6 (2)
C(20)	0.6185 (8)	0.1096 (7)	0.1219 (4)	5.9 (2)
C(21)	0.5785 (9)	0.1203 (7)	0.0559 (4)	5.6 (2)
C(22)	0.4257 (8)	0.1717 (6)	0.0402 (3)	4.3 (1)
C(23)	0.374 (1)	0.1875 (7)	−0.0284 (3)	5.7 (2)
C(24)	0.2279 (9)	0.2393 (7)	−0.0433 (3)	5.3 (2)
C(25)	0.1172 (8)	0.2796 (6)	0.0103 (3)	4.3 (1)
C(26)	−0.0357 (9)	0.3338 (7)	−0.0035 (4)	5.5 (2)
C(27)	−0.1401 (9)	0.3699 (8)	0.0491 (4)	5.6 (2)
C(28)	−0.0901 (8)	0.3502 (7)	0.1150 (4)	4.8 (2)
C(29)	0.1593 (7)	0.2644 (5)	0.0785 (3)	3.4 (1)
C(30)	0.3172 (7)	0.2099 (5)	0.0939 (3)	3.4 (1)
C(31)	0.410 (1)	0.4891 (8)	0.1659 (5)	6.3 (2)
C(32)	0.429 (1)	0.541 (1)	0.0910 (5)	8.6 (3)
Complex (2)				
Sm	0.13189 (4)	0.24687 (3)	0.26288 (2)	2.701 (5)
Cl(1)	0.0509 (3)	−0.0426 (2)	0.08837 (9)	5.93 (5)
Cl(2)	0.2754 (3)	−0.1679 (2)	0.1860 (1)	7.39 (5)
Cl(3)	−0.3258 (3)	0.7301 (2)	0.2269 (1)	5.94 (5)
Cl(4)	−0.1697 (3)	0.6449 (2)	0.3532 (1)	6.59 (5)
Cl(5)	0.3377 (5)	0.5838 (2)	0.3402 (2)	11.7 (1)
Cl(6)†	0.4460 (8)	0.4382 (5)	0.4530 (2)	10.7 (1)
Cl(6′)†	0.3136 (5)	0.3895 (3)	0.4623 (2)	5.10 (9)
O(1)	0.1119 (5)	0.0566 (4)	0.2292 (2)	3.80 (9)
O(2)	−0.1298 (6)	−0.0038 (4)	0.2430 (3)	5.4 (1)
O(3)	−0.0354 (6)	0.4340 (4)	0.2686 (2)	4.1 (1)
O(4)	−0.0072 (6)	0.6052 (4)	0.1887 (3)	5.1 (1)
O(5)	0.3573 (5)	0.2860 (4)	0.3098 (2)	4.1 (1)
O(6)	0.5798 (6)	0.3437 (5)	0.3310 (3)	5.9 (1)
O(7)	−0.1490 (5)	0.2101 (4)	0.2808 (2)	3.76 (9)
O(8)	0.2543 (6)	0.4399 (5)	0.1855 (3)	5.4 (1)
N(1)	0.0342 (6)	0.2318 (4)	0.3993 (2)	3.2 (1)
N(2)	0.2514 (6)	0.0549 (4)	0.3535 (3)	3.3 (1)
N(3)	0.3562 (6)	0.1963 (5)	0.1657 (3)	3.4 (1)
N(4)	0.0504 (6)	0.2951 (5)	0.1363 (3)	3.3 (1)
C(1)	0.0134 (7)	−0.0093 (5)	0.2217 (3)	3.1 (1)
C(2)	0.0743 (8)	−0.1099 (6)	0.1785 (3)	3.9 (1)
C(3)	−0.0770 (8)	0.5437 (6)	0.2406 (3)	3.4 (1)
C(4)	−0.2277 (8)	0.6007 (6)	0.2800 (3)	3.6 (1)
C(5)	0.4345 (7)	0.3460 (5)	0.3356 (3)	3.3 (1)
C(6)	0.325 (1)	0.4348 (7)	0.3768 (4)	5.4 (2)
C(7)	−0.0792 (8)	0.3117 (6)	0.4225 (3)	3.8 (1)
C(8)	−0.1219 (9)	0.3132 (7)	0.4935 (4)	4.7 (2)
C(9)	−0.0415 (9)	0.2300 (7)	0.5423 (3)	4.6 (2)
C(10)	0.0781 (8)	0.1423 (6)	0.5207 (3)	3.7 (1)
C(11)	0.1644 (9)	0.0501 (7)	0.5688 (4)	4.9 (2)
C(12)	0.2736 (9)	−0.0338 (7)	0.5462 (4)	4.9 (2)
C(13)	0.3098 (8)	−0.0361 (6)	0.4730 (4)	3.9 (1)
C(14)	0.4211 (9)	−0.1230 (7)	0.4474 (4)	5.1 (2)
C(15)	0.444 (1)	−0.1225 (7)	0.3772 (4)	5.3 (2)
C(16)	0.3538 (8)	−0.0328 (6)	0.3327 (4)	4.3 (2)
C(17)	0.2282 (7)	0.0541 (5)	0.4238 (3)	3.0 (1)
C(18)	0.1124 (7)	0.1461 (5)	0.4474 (3)	3.0 (1)
C(19)	0.5050 (8)	0.1497 (7)	0.1781 (4)	4.5 (2)
C(20)	0.6194 (9)	0.1095 (8)	0.1277 (5)	5.8 (2)
C(21)	0.579 (1)	0.1201 (7)	0.0616 (4)	5.8 (2)
C(22)	0.4262 (8)	0.1706 (6)	0.0452 (4)	4.2 (2)
C(23)	0.376 (1)	0.1868 (7)	−0.0242 (4)	5.5 (2)
C(24)	0.230 (1)	0.2381 (7)	−0.0380 (4)	5.3 (2)
C(25)	0.1169 (9)	0.2781 (6)	0.0148 (3)	4.3 (2)
C(26)	−0.038 (1)	0.3336 (8)	0.0013 (4)	5.4 (2)
C(27)	−0.144 (1)	0.3677 (8)	0.0540 (4)	5.5 (2)
C(28)	−0.0943 (8)	0.3468 (7)	0.1213 (4)	4.4 (2)
C(29)	0.1580 (7)	0.2609 (5)	0.0839 (3)	3.2 (1)

C(30)	0.3169 (7)	0.2076 (5)	0.0995 (3)	3.3 (1)
C(31)	0.410 (1)	0.4818 (8)	0.1658 (5)	5.9 (2)
C(32)	0.425 (1)	0.540 (1)	0.0914 (5)	7.9 (3)

† Site occupancy = 0.5.

Table 2. *Selected bond lengths* (Å) *and angles* (°)

Complex (1)		Complex (2)	
La—O(1)	2.486 (3)	Sm—O(1)	2.422 (3)
La—O(3)	2.462 (3)	Sm—O(3)	2.379 (3)
La—O(5)	2.462 (3)	Sm—O(5)	2.369 (3)
La—O(7)	2.535 (3)	Sm—O(7)	2.443 (3)
La—O(8)	2.659 (3)	Sm—O(8)	2.629 (4)
La—N(1)	2.749 (3)	Sm—N(1)	2.667 (3)
La—N(2)	2.735 (4)	Sm—N(2)	2.650 (4)
La—N(3)	2.706 (3)	Sm—N(3)	2.594 (4)
La—N(4)	2.698 (4)	Sm—N(4)	2.591 (3)
O(1)—La—O(3)	136.1 (1)	O(1)—Sm—O(3)	137.0 (1)
O(1)—La—O(5)	122.8 (1)	O(1)—Sm—O(5)	124.7 (1)
O(1)—La—O(7)	70.14 (9)	O(1)—Sm—O(7)	70.8 (1)
O(1)—La—O(8)	130.1 (1)	O(1)—Sm—O(8)	127.9 (1)
O(1)—La—N(1)	108.2 (1)	O(1)—Sm—N(1)	110.2 (1)
O(1)—La—N(2)	67.9 (1)	O(1)—Sm—N(2)	68.2 (1)
O(1)—La—N(3)	67.2 (1)	O(1)—Sm—N(3)	67.2 (1)
O(1)—La—N(4)	71.2 (1)	O(1)—Sm—N(4)	71.1 (1)
O(3)—La—O(5)	100.8 (1)	O(3)—Sm—O(5)	98.8 (1)
O(3)—La—O(7)	69.2 (1)	O(3)—Sm—O(7)	70.7 (1)
O(3)—La—O(8)	66.5 (1)	O(3)—Sm—O(8)	66.5 (1)
O(3)—La—N(1)	75.6 (1)	O(3)—Sm—N(1)	75.3 (1)
O(3)—La—N(2)	135.1 (1)	O(3)—Sm—N(2)	136.8 (1)
O(3)—La—N(3)	129.8 (1)	O(3)—Sm—N(3)	129.2 (1)
O(3)—La—N(4)	83.6 (1)	O(3)—Sm—N(4)	82.1 (1)
O(5)—La—O(7)	150.1 (1)	O(5)—Sm—O(7)	148.7 (1)
O(5)—La—O(8)	69.7 (1)	O(5)—Sm—O(8)	68.7 (1)
O(5)—La—N(1)	75.9 (1)	O(5)—Sm—N(1)	75.9 (1)
O(5)—La—N(2)	66.7 (1)	O(5)—Sm—N(2)	67.7 (1)
O(5)—La—N(3)	81.8 (1)	O(5)—Sm—N(3)	80.5 (1)
O(5)—La—N(4)	132.6 (1)	O(5)—Sm—N(4)	132.4 (1)
O(7)—La—O(8)	125.5 (1)	O(7)—Sm—O(8)	127.3 (1)
O(7)—La—N(1)	74.3 (1)	O(7)—Sm—N(1)	72.9 (1)
O(7)—La—N(2)	99.8 (1)	O(7)—Sm—N(2)	99.3 (1)
O(7)—La—N(3)	126.8 (1)	O(7)—Sm—N(3)	129.4 (1)
O(7)—La—N(4)	75.8 (1)	O(7)—Sm—N(4)	76.7 (1)
O(8)—La—N(1)	121.5 (1)	O(8)—Sm—N(1)	121.7 (1)
O(8)—La—N(2)	134.2 (1)	O(8)—Sm—N(2)	133.1 (1)
O(8)—La—N(3)	67.8 (1)	O(8)—Sm—N(3)	66.2 (1)
O(8)—La—N(4)	69.2 (1)	O(8)—Sm—N(4)	68.4 (1)
N(1)—La—N(2)	59.6 (1)	N(1)—Sm—N(2)	61.7 (1)
N(1)—La—N(3)	149.4 (1)	N(1)—Sm—N(3)	148.6 (1)
N(1)—La—N(4)	148.1 (1)	N(1)—Sm—N(4)	146.7 (1)
N(2)—La—N(3)	92.4 (1)	N(2)—Sm—N(3)	90.4 (1)
N(2)—La—N(4)	137.6 (1)	N(2)—Sm—N(4)	138.0 (1)
N(3)—La—N(4)	61.4 (1)	N(3)—Sm—N(4)	64.0 (1)

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

D—H···*A*	*D*···*A*	*D*—H···*A*
Complex (1)		
O(7)—H(71)···O(2)	2.680 (7)	165.1 (2)
O(7)—H(72)···O(6^{i})	2.725 (6)	166.5 (2)
O(8)—H(08)···O(4)	2.718 (6)	160.4 (2)
Complex (2)		
O(7)—H(71)···O(2)	2.658 (7)	161.1 (2)
O(7)—H(72)···O(6^{i})	2.743 (6)	166.7 (2)
O(8)—H(08)···O(4)	2.679 (7)	155.2 (2)

Symmetry code: (i) $x - 1, y, z$.

The data were corrected for Lorentz and polarization effects. All non-H atoms were located through Patterson and Fourier techniques and were refined by full-matrix least-squares methods including anisotropic displacement parameters. The atom Cl(6) of one dichloroacetato group was originally treated as fully occupied, but a strong peak along the C—Cl(6) vector was then found in the difference Fourier map, and was included in the model as atom Cl(6′); the atoms Cl(6) and Cl(6′) were assigned a site occupancy of 0.5 each. 19 H atoms of complex (1) and 21 H atoms of complex (2) were located by the same method but were not refined. Program used throughout the analysis: Enraf–Nonius *SDP-Plus* (Frenz, 1985).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, bond distances and angles involving H atoms, least-squares-planes data and hydrogen-bond data have been deposited with the IUCr (Reference: AB1156). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis[tris(2-hydroxyethyl)amine]nickel(II) Chloride

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Abstract

The crystal structure of bis(2,2′,2″-nitrilotriethanol-*N*,*O*,*O*′)nickel(II) chloride, $[Ni(C_6H_{15}NO_3)_2]Cl_2$, has been determined from X-ray intensity data. The structure consists of an $[Ni(C_6H_{15}NO_3)_2]^{2+}$ cation and two

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