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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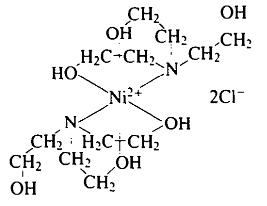
Cl^- anions. The central Ni atom in the cation is coordinated by two 2,2′,2″-nitrilotriethanol ligands, with bonds to four O atoms and two N atoms resulting in a distorted octahedral environment. The N atoms occupy *trans* positions and the O3 atom does not coordinate the Ni atom. The Cl anions form three strong intermolecular hydrogen bonds with the hydroxy H atoms of different cations.

Comment

In the course of the synthesis of transition metal complexes of tris(2-hydroxyethyl)amine (alternative names 2,2′,2″-nitrilotriethanol and triethanolamine), blue single crystals of the Ni^{II} complex, (I), were prepared for structure analysis.



(I)

The central Ni atom in the complex cation is octahedrally coordinated by the two effectively tridentate ligands. Each ligand supplies one N atom and two O atoms for bonding, with the N atoms occupying *trans* positions in the coordination polyhedron. The O3 atom does not coordinate to the Ni atom (Fig. 1).

The Ni—O1 and Ni—O2 distances in the complex are 2.068 (1) and 2.067 (1) Å, respectively. The Ni—O distances are significantly shorter than those in other O- and N-donor octahedral complexes of Ni^{II} [*e.g.* 2.08 (1) Å reported by Castellano, Nascimento & Calvo (1982) and 2.103 (4) Å by Garcia-Granda & Gomez-Beltran (1984)] and are somewhat longer than in $[Ni(C_5HF_6O_2)(C_{18}H_{15}OP)_2]$ [2.049 (3) Å; Polam & Porter, 1992], and are comparable with those found in $C_{24}H_{27}N_3O_5S_2Ni$ [2.070 (6) Å; Garnovskii *et al.*, 1994]. The Ni—N distance is 2.113 (1) Å, which is similar to the average Ni—N distance found for the octahedral complex $[Ni(NCS)(C_3H_4N_2)_4]$ [2.113 (4) Å; Koman, Jona & Maslejova, 1991] and smaller than that of dichlorotetrakis(pyridine)nickel(II) [2.121 (3) Å; Bachman, Whitmire, Mandal & Bharadwaj, 1992]. The O2—Ni—O1, N—Ni—O1 and N—Ni—O2 angles are 91.31 (6), 83.64 (6) and 81.69 (6)°, respectively. The deviation of the interaxial angles from the theoretical value of a perfect octahedron (90°) shows the significant distortion in the complex. Thus, the coordination geometry about the Ni^{II} atom is best described as distorted octahedral.

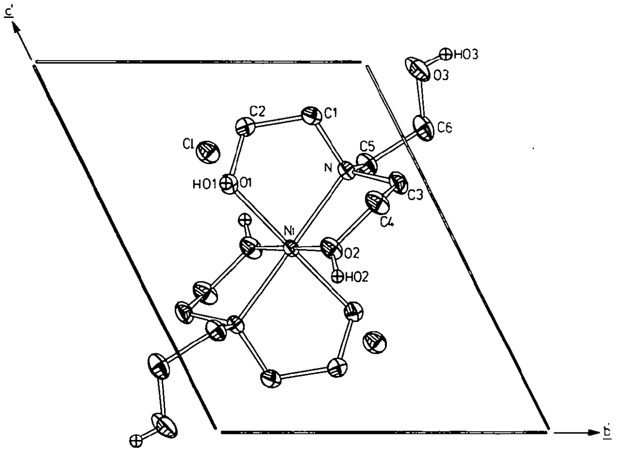


Fig. 1. The unit cell of the title complex viewed down the *a* axis. Displacement ellipsoids are shown at the 40% probability level for non-H atoms. Hydroxyl H atoms are represented by small spheres of arbitrary radii and methylene H atoms have been omitted for clarity.

The two Cl atoms in the structure are not coordinated to the metal but instead form strong intermolecular hydrogen bonds with the hydroxyl H atoms of three different complex cations: HO1···Cl 2.34 (4), $HO2\cdots Cl^{i}$ 2.23 (4) and $HO3\cdots Cl^{ii}$ 2.48 (4) Å [symmetry codes: (i) $2-x, 1-y, 1-z$; (ii) $1-x, 2-y, 2-z$]. The interaction of the Cl^- ions with the HO2 atom is much stronger and results in the lengthening of the covalent O—H bond (Table 3) as explained by Steiner & Saenger (1994).

Experimental

Bis[tris(2-hydroxyethyl)amine]nickel(II) chloride was prepared by reaction of anhydrous nickel(II) chloride and triethanolamine in isobutanol. Blue single crystals were obtained by slow evaporation of the solvent at room temperature.

Crystal data

$[Ni(C_6H_{15}NO_3)_2]Cl_2$
$M_r = 427.995$
Triclinic
$P\bar{1}$
$a = 7.374 (4)$ Å
$b = 7.469 (4)$ Å
$c = 9.598 (4)$ Å
$\alpha = 117.57 (3)°$
$\beta = 72.19 (4)°$
$\gamma = 96.81 (4)°$
$V = 445.9 (4)$ Å^3
$Z = 1$
$D_x = 1.593$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 14 reflections
$\theta = 10.5$–$14.6°$
$\mu = 1.42$ mm^{-1}
$T = 293$ K
Block
0.6 × 0.5 × 0.4 mm
Blue

Data collection

Nicolet *P*3 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2628 measured reflections
2585 independent reflections
2434 observed reflections [$I > 2\sigma(I)$]

$R_{int} = 0.045$
$\theta_{max} = 30°$
$h = -9 \rightarrow 10$
$k = -10 \rightarrow 9$
$l = 0 \rightarrow 13$
2 standard reflections
frequency: 50 min
intensity decay: 2.5%

Refinement

Refinement on *F*
$R = 0.038$
$wR = 0.044$
2434 reflections
117 parameters
$w = 1/[\sigma^2(F) + 0.003352F^2]$
$(\Delta/\sigma)_{\max} = 0.032$
$\Delta\rho_{\max} = 1.4$ e Å^{-3}
$\Delta\rho_{\min} = -1.3$ 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)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Ni	1/2	1/2	1/2	0.0175 (1)
Cl	0.86552 (6)	0.39203 (7)	0.75640 (6)	0.0323 (1)
O1	0.5051 (2)	0.4076 (2)	0.6713 (2)	0.0254 (3)
O2	0.7674 (2)	0.6204 (2)	0.4991 (2)	0.0290 (3)
O3	0.2030 (3)	1.1479 (2)	0.9817 (2)	0.0446 (5)
N	0.4173 (2)	0.7825 (2)	0.7075 (2)	0.0205 (3)
C1	0.4154 (3)	0.7593 (3)	0.8547 (2)	0.0263 (4)
C2	0.3784 (2)	0.5427 (3)	0.8249 (2)	0.0269 (4)
C3	0.5690 (3)	0.9187 (2)	0.6746 (2)	0.0286 (4)
C4	0.7649 (3)	0.8256 (3)	0.6212 (2)	0.0334 (5)
C5	0.2262 (2)	0.8510 (3)	0.7236 (2)	0.0269 (4)
C6	0.1811 (3)	1.0763 (3)	0.8222 (3)	0.0344 (5)

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

Ni—O1	2.068 (1)	O1—C2	1.438 (2)
Ni—O2	2.067 (1)	O2—C4	1.433 (2)
Ni—N	2.113 (1)	O3—C6	1.424 (3)
C1—N	1.496 (2)	C1—C2	1.513 (2)
C3—N	1.482 (2)	C3—C4	1.513 (3)
C5—N	1.495 (2)	C5—C6	1.518 (2)
N—Ni—O1	83.64 (6)	C1—N—Ni	107.6 (1)
N—Ni—O2	81.69 (6)	C3—N—Ni	103.7 (1)
O1—Ni—O2	91.31 (6)	C5—N—Ni	110.9 (1)
C2—O1—Ni	106.8 (1)	C1—N—C5	111.2 (1)
C4—O2—Ni	113.9 (1)	C3—N—C5	111.5 (1)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—HO1···Cl	0.72 (4)	2.34 (4)	3.039 (2)	164 (4)
O2—HO2···Cl$^{\rm i}$	0.81 (4)	2.23 (4)	3.023 (2)	167 (4)
O3—HO3···Cl$^{\rm ii}$	0.71 (4)	2.48 (4)	3.166 (2)	161 (4)

Symmetry codes: (i) $2 - x, 1 - y, 1 - z$; (ii) $1 - x, 2 - y, 2 - z$.

Data collection used 2θ scan rates of 5.33 ($I_p < 150$) to 58.6° min^{-1} ($I_p > 2500$), where I_p is the prescan intensity. Scan widths were 2.4 to 2.8° (2θ). Refinement was by full-matrix least squares. All non-H atoms were refined anisotropically. The initial positions of hydroxyl H atoms were determined by rigid-body refinement of O-centred 'methyl' groups, each with two dummy H atoms having zero site-occupancy factors, and then refined in the usual manner but with a common group $U_{\rm iso}$. Methylene H atoms were placed in calculated positions (C—H 0.95 Å) and refined riding on the C atom to which they were attached with a second common group $U_{\rm iso}$. All computations were performed on the SUN SPARCserver (UNIX operating system) of the Computing Centre of the University of Aberdeen.

Data reduction: *RDNIC* (Howie, 1980). Structure solution: *SHELXS*86 (Sheldrick, 1990). Structure refinement: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *PLOTAID* (Cradwick, 1980).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HR1050). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Trichloro[tris(2-pyridyl)methanol]-rhodium(III)–Methanol (1/1)

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Abstract

An X-ray diffraction study of [Rh{$(C_5H_4N)_3COH$}Cl_3].-CH_3OH showed that the tris(2-pyridyl)methanol ligand coordinates to the Rh atom in a tridentate fashion, with the N atoms positioned at three corners of an octahedron