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Diaquatris(nicotinato-*κO*)bis(1,10-phenanthroline-κ^2*N*,*N′*)neodymium(III) trihydrate

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Key indicators

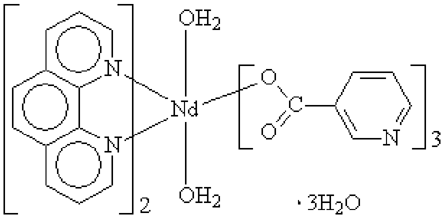
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.029
wR factor = 0.061
Data-to-parameter ratio = 14.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $[Nd(C_6H_4NO_2)_3(C_{12}H_8N_2)_2(H_2O)_2]\cdot 3H_2O$, consists of a mononuclear $[Nd(C_6H_4NO_2)_3(C_{12}H_8N_2)_2(H_2O)_2]$ molecule and three uncoordinated water molecules. The Nd^{III} atom is nine-coordinate through coordination by the O atoms of three nicotinate groups, four N atoms of two phenanthroline molecules and two water molecules in a tricapped trigonal prismatic coordination geometry. The mononuclear molecule and solvent water molecule are linked by hydrogen bonds to form a layer framework.

Comment

A number of crystal structures of lanthanide compounds having 1,10-phenanthroline and carboxylate groups have been reported, such as tetra-benzoatobis[benzoato(phenanthroline)lanthanum] (Shi *et al.*, 2001), dimethacrylatobis-[dimethacrylato(phenanthroline)ytterbium] (Lu *et al.*, 2000) and dipivalatobis(phenanthroline)nitratopraseodymium (Pisarevskii *et al.*, 1995). The nicotinate anion has been widely used in metal-organic complexes (Hao *et al.*, 2000; Lin *et al.*, 1998) but not used in a lanthanide complex having phenanthroline other than in diaqua(phenanthroline)trinicotinato-europium dimethyl sulfoxide solvate (Palkina *et al.*, 1995) and the praseodymium analog (Yue *et al.*, 2004).



(I)

The asymmetric unit of the crystal structure of the title Nd complex, (I), consists of mononuclear $[Nd(C_6H_4NO_2)_3(C_{12}H_8N_2)_2(H_2O)_2]$ and three uncoordinated water molecules. The Nd atom is nine-coordinate through coordination by the O atoms of three nicotinate groups, four N-atom donors of two phenanthroline molecules and two water molecules in a tricapped trigonal prismatic coordination geometry (Fig. 1). The bond dimensions compare well with those found in reported analogs. Hydrogen bonds link the molecules into a two-dimensional structure (Table 2).

Experimental

A solution of nicotinic acid (0.037 g, 0.3 mmol) in water (10 ml) was mixed with an aqueous solution of $NdCl_3\cdot 6H_2O$ (0.036 g, 0.1 mmol) and a solution of phenanthroline (0.054 g, 0.3 mmol) in EtOH (25 ml). The mixture was filtered and the filtrate was left to stand at room temperature. Crystals suitable for X-ray analysis were obtained after 3 d.

Crystal data

$[Nd(C_6H_4NO_2)_3(C_{12}H_8N_2)_2$-
$(H_2O)_2]\cdot 3H_2O$
$M_r = 961.03$
Triclinic, $P\overline{1}$
$a = 9.066$ (3) Å
$b = 12.924$ (4) Å
$c = 17.854$ (6) Å
$\alpha = 84.894$ (5)°
$\beta = 80.659$ (5)°
$\gamma = 84.306$ (5)°
$V = 2048.3$ (11) Å^3

$Z = 2$
$D_x = 1.558$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 989 reflections
$\theta = 3.3$–26.7°
$\mu = 1.34$ mm^{-1}
$T = 293$ (2) K
Block, colorless
0.20 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996; Blessing, 1995)
$T_{\min} = 0.776$, $T_{\max} = 0.795$
11882 measured reflections

8297 independent reflections
7240 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.018$
$\theta_{\max} = 26.4°$
$h = -6 \rightarrow 11$
$k = -16 \rightarrow 16$
$l = -22 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.061$
$S = 1.08$
8297 reflections
590 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0151P)^2 + 1.5345P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.73$ e Å^{-3}
$\Delta\rho_{\min} = -0.58$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Nd1—O2	2.475 (2)	Nd1—N4	2.722 (2)
Nd1—O3	2.363 (2)	Nd1—N5	2.698 (3)
Nd1—O5	2.483 (2)	Nd1—N6	2.728 (2)
Nd1—O7	2.488 (2)	Nd1—N7	2.695 (2)
Nd1—O8	2.476 (2)		
O2—Nd1—O3	78.31 (7)	O5—Nd1—N5	68.64 (7)
O2—Nd1—O5	138.32 (6)	O5—Nd1—N6	75.98 (7)
O2—Nd1—O7	70.55 (7)	O5—Nd1—N7	136.29 (7)
O2—Nd1—O8	139.79 (7)	O7—Nd1—O8	69.41 (8)
O2—Nd1—N4	68.65 (7)	O7—Nd1—N4	114.38 (8)
O2—Nd1—N5	91.88 (7)	O7—Nd1—N5	72.09 (8)
O2—Nd1—N6	130.69 (7)	O7—Nd1—N6	118.60 (8)
O2—Nd1—N7	78.83 (7)	O7—Nd1—N7	75.20 (8)
O3—Nd1—O5	88.00 (7)	O8—Nd1—N4	133.46 (8)
O3—Nd1—O7	141.55 (8)	O8—Nd1—N5	79.41 (8)
O3—Nd1—O8	135.31 (8)	O8—Nd1—N6	68.69 (8)
O3—Nd1—N4	72.36 (7)	O8—Nd1—N7	87.62 (8)
O3—Nd1—N5	132.04 (7)	N4—Nd1—N5	60.51 (7)
O3—Nd1—N6	67.24 (7)	N4—Nd1—N6	127.02 (7)
O3—Nd1—N7	77.16 (7)	N4—Nd1—N7	138.92 (7)
O5—Nd1—O7	130.36 (8)	N5—Nd1—N6	137.40 (7)
O5—Nd1—O8	74.53 (7)	N5—Nd1—N7	147.24 (8)
O5—Nd1—N4	69.70 (7)	N6—Nd1—N7	60.33 (7)

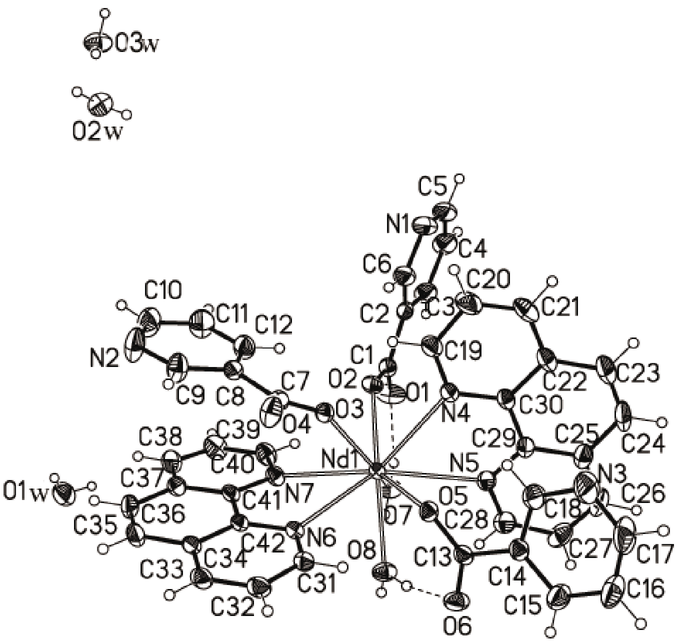


Figure 1
The molecular structure of the title compound, shown with 30% probability displacement ellipsoids. Dashed lines indicate hydrogen bonds.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O7—H7a···O1	0.81 (1)	1.85 (2)	2.627 (3)	160 (4)
O7—H7b···O2$w^{\rm i}$	0.82 (1)	1.97 (2)	2.758 (3)	161 (4)
O8—H8a···O5	0.82 (1)	2.62 (4)	3.002 (3)	110 (3)
O8—H8a···O6	0.82 (1)	1.81 (1)	2.617 (3)	167 (4)
O8—H8b···O3$w^{\rm i}$	0.82 (1)	1.89 (1)	2.711 (4)	179 (4)
O1w—H1wa···N1$^{\rm i}$	0.82 (1)	2.08 (2)	2.868 (5)	162 (5)
O1w—H1wb···N2$^{\rm ii}$	0.81 (1)	2.10 (2)	2.900 (5)	167 (5)
O2w—H2wa···O4$^{\rm iii}$	0.82 (1)	1.91 (1)	2.727 (4)	176 (4)
O2w—H2wb···O5$^{\rm iii}$	0.82 (1)	2.10 (2)	2.898 (3)	164 (4)
O3w—H3wa···O2w	0.81 (1)	2.00 (2)	2.781 (4)	161 (4)
O3w—H3wb···O1$w^{\rm iv}$	0.81 (1)	1.97 (1)	2.773 (4)	172 (4)

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

H atoms attached to C atoms were included in calculated positions and treated as riding atoms [C—H = 0.93 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$]. Water H atoms were located in Fourier difference maps and refined isotropically, with an O—H distance restraint of 0.82 (1) Å.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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