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Triaquatris[tetraoxorhenato(VII)]neodymium(III)

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The title compound, $Nd(ReO_4)_3(H_2O)_3$, crystallizes isostructurally with its analogous triaquatris[tetraoxorhenato(VII)]-lanthanum(III) compound. The neodymium is coordinated by three water molecules and six O atoms of six tetraoxorhenate(VII) anions. The coordination polyhedron at the Nd is that of a tricapped trigonal prism. The three coordinated water molecules form five hydrogen bonds to four tetraoxorhenate(VII) anions.

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I thank Thomas Hecht for the preparation of the title compound, during a course in advanced inorganic chemistry.

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(Nd–O) = 0.007 Å
R factor = 0.024
wR factor = 0.055
Data-to-parameter ratio = 11.0

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

Comment

Rare earth tetraoxorhenates(VII) are known as precursors for the synthesis of mixed rare earth oxides (Mujica *et al.*, 1999). During the course of an investigation of the reactivity of rhenium metal with oxidizing agents we found that the *in situ* generated 'perrhenic acid' readily reacts with neodymium(III) oxide to form triaquatris[tetraoxorhenato(VII)]neodymium(III), (I). The title compound crystallizes in the centrosymmetric space group $P2_1/c$ with one neodymium, three crystallographically independent tetraoxorhenate(VII) anions and three water molecules in the asymmetric unit. This compound is configurationally isotypic with triaquatris-[tetraoxorhenato(VII)]lanthanum(III) (Mujica *et al.*, 1997).

Accordingly, the neodymium is ninefold coordinated by three water molecules and six tetraoxorhenate(VII) anions to form the coordination figure of a distorted tricapped trigonal prism. The vertices of the trigonal prism are occupied by four O atoms of four ReO_4^- anions and the two O atoms of the water ligands, while the three capped positions are occupied by two ReO_4^- O atoms and the O atom of one water molecule (Fig. 1).

This coordination mode is comparable with those of the well known rare earth nonaaqua complexes $[Ln(H_2O)_9$-$(CF_3SO_3)_3$; Ln = trivalent rare earth metal cation; Harrowfield *et al.*, 1983] and the isotypic bismuth compound (Frank *et al.*, 1995). Due to the high symmetry of these simple aqua complex cations the neodymium–water distances of the six symmetry-equivalent water molecules occupying the vertices of the trigonal prism are 2.451 (2) and 2.571 (2) Å for the three symmetry equivalent water molecules of the capped positions (Chatterjee *et al.*, 1988).

In the structure of the title compound, there is a coordination polyhedron of a distorted three capped trigonal prism for the neodymium cation, with the Nd—O distances varying in the range 2.470 (6)–2.543 (4) Å. But in contrast to the pure aqua complexes discussed above there are shorter and longer Nd—O distances found for the O atoms occupying the vertices of the trigonal prism as well as those of the capped positions (Table 1).

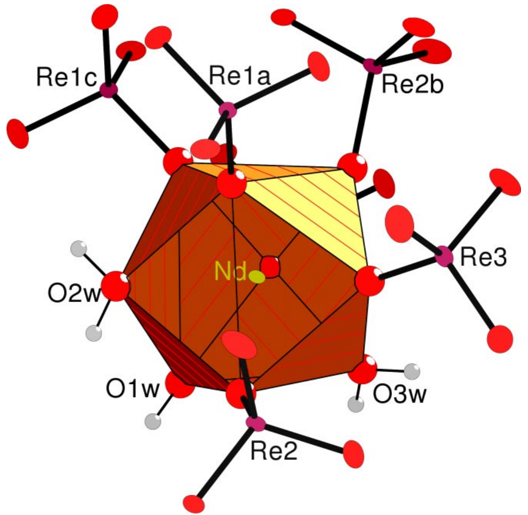

Figure 1
Each neodymium has a coordination polyhedron of a distorted tricapped trigonal prism built by six tetraoxorhenate(VII) anions and three water molecules. The ellipsoids are drawn at the 50% probability level. Atoms used to build the polyhedron are drawn with an arbitrary radius [symmetry code: (*a*) $1 + x, y, z$; (*b*) $1 - x, -\frac{1}{2} + y, \frac{1}{2} - z$; (*c*) $-x, -y, -z$].

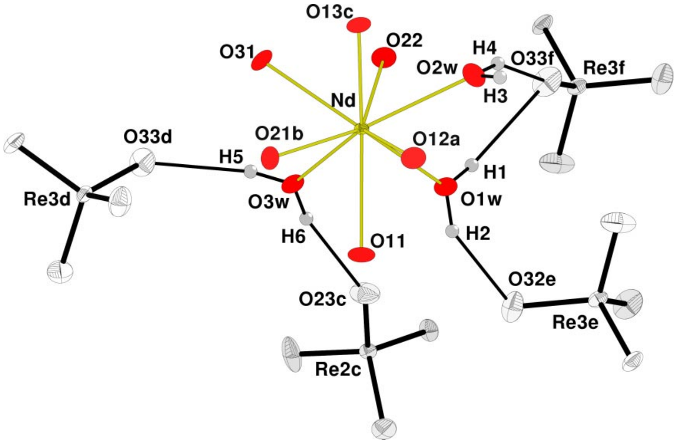

Figure 2
The coordination of the neodymium centre and the hydrogen-bonding motif of the three water ligands. The ellipsoids are drawn at the 50% probability level [symmetry code: (*a*) $-x, -y, -z$; (*b*) $1 - x, -\frac{1}{2} + y, \frac{1}{2} - z$; (*c*) (*c*) $1 + x, y, z$; (*d*) $1 - x, -y, -1 - z$; (*e*) $1 + x, \frac{1}{2} - y, \frac{1}{2} + z$; (*f*) $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$].

The three water ligands at the neodymium form five weak hydrogen bonds to four neighbouring tetraoxorhenate(VII) anions (Fig. 2 and Table 2). The three-dimensional framework built by the anions coordinating the neodymium centre is best described as $\mathrm{Nd(ReO_4)(ReO_4)_{2/2}(ReO_4)_{3/3}}$, while the Re—O distances of the $\mathrm{ReO_4^-}$ anions vary in the range 1.712 (5)–1.728 (5) Å (Fig. 3).

Experimental

Neodymium(III) oxide readily reacts with perrhenic acid, which was *in situ* generated by the reaction of rhenium powder and H_2O_2, to yield a colourless solution. From this solution very thin platelets can be grown within a few days at room temperature.

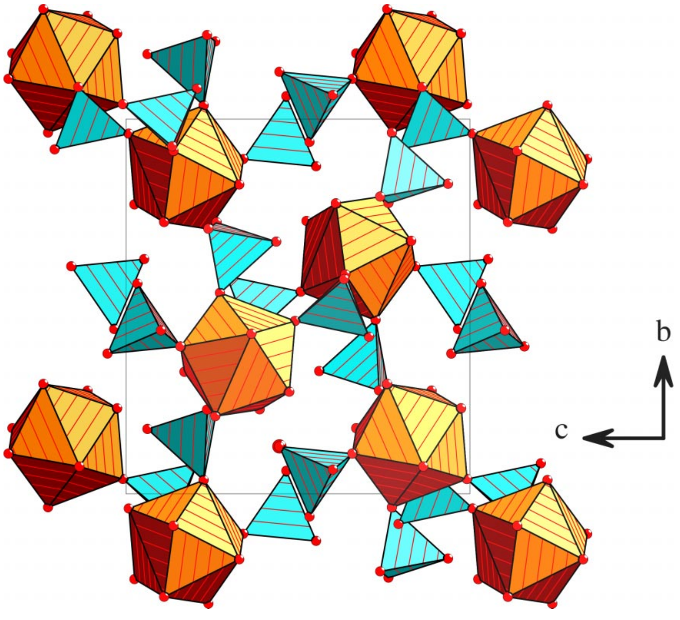

Figure 3
Packing of the title compound with a view along [$\bar{1}$00].

Crystal data

$\mathrm{Nd(ReO_4)_3(H_2O)_3}$
$M_r = 948.89$
Monoclinic, $P2_1/c$
$a = 7.550$ (2) Å
$b = 13.660$ (3) Å
$c = 12.850$ (3) Å
$\beta = 102.90$ (3)°
$V = 1291.8$ (5) Å^3
$Z = 4$

$D_x = 4.879$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5000 reflections
$\theta = 2.8$–25.0°
$\mu = 32.05$ mm^{-1}
$T = 293$ (2) K
Platelet, colourless
0.50 × 0.10 × 0.01 mm

Data collection

Stoe IPDS diffractometer
ω scans
Absorption correction: numerical (*XRED*; Stoe & Cie, 1996)
$T_{\min} = 0.026$, $T_{\max} = 0.680$
6587 measured reflections
2144 independent reflections

1915 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.068$
$\theta_{\max} = 25.0°$
$h = -8 \rightarrow 8$
$k = -16 \rightarrow 15$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.024$
$wR(F^2) = 0.055$
$S = 1.02$
2144 reflections
195 parameters
H atoms refined with geometrical restraints

$w = 1/[\sigma^2(F_o^2) + (0.024P)^2 + 2.400P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.188$
$\Delta\rho_{\max} = 1.14$ e Å^{-3}
$\Delta\rho_{\min} = -1.30$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.00057 (7)

Table 1
Selected bond lengths (Å).

Nd1—O11	2.543 (4)	Nd1—O3*W*	2.470 (6)
Nd1—O22	2.494 (6)	Nd1—O12$^{\rm i}$	2.476 (5)
Nd1—O31	2.479 (5)	Nd1—O21$^{\rm ii}$	2.483 (5)
Nd1—O1*W*	2.484 (6)	Nd1—O13$^{\rm iii}$	2.520 (4)
Nd1—O2*W*	2.486 (7)		

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

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1W—H1$W\cdots$O33$^{\rm i}$	0.90 (1)	2.27 (6)	3.073 (11)	148 (10)
O1W—H2$W\cdots$O32$^{\rm ii}$	0.90 (1)	2.29 (3)	3.009 (10)	137 (3)
O2W—H4$W\cdots$O33$^{\rm i}$	0.90 (1)	2.08 (3)	2.954 (10)	163 (6)
O3W—H5$W\cdots$O33$^{\rm iii}$	0.90 (1)	2.17 (5)	3.024 (11)	158 (12)
O3W—H6$W\cdots$O23$^{\rm iv}$	0.90 (1)	1.99 (3)	2.851 (9)	159 (4)

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

The atomic coordinates of the H atoms were refined using O—H and H—H distance restraints. One common $U_{\rm iso}$ value for all H atoms of the water molecules has been refined.

Data collection: *IPDS Software* (Stoe & Cie, 1998); cell refinement: *IPDS Software*; data reduction: *IPDS Software*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 1998); software used to prepare material for publication: *SHELXL*97.

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