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#### Key indicators

Single-crystal X-ray study

$T = 293$  K

Mean  $\sigma(C-C) = 0.007$  Å

Disorder in main residue

$R$  factor = 0.034

$wR$  factor = 0.077

Data-to-parameter ratio = 13.6

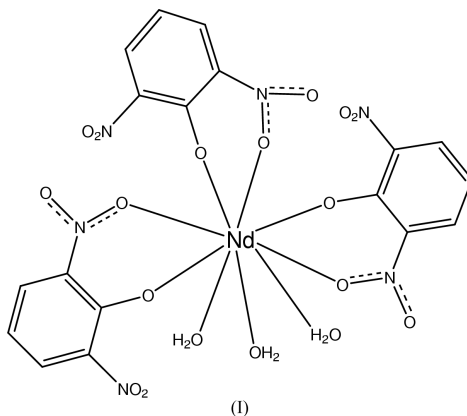
For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.

## Triaquatis(2,6-dinitrophenolato)neodymium(III)

The title compound,  $[\text{Nd}(\text{C}_6\text{H}_3\text{N}_2\text{O}_5)_3(\text{H}_2\text{O})_3]$ , has a nine-coordinated system with a slightly distorted tricapped trigonal prismatic coordination geometry. The  $\text{Nd}^{\text{III}}$  ion is coordinated to three  $\text{H}_2\text{O}$  and by three bidentate 2,6-dinitrophenolate ligands. The  $\text{Nd}-\text{O}$  bond distances are in the range 2.311 (3)–2.730 (3) Å. The  $\text{Nd}-\text{O}$  distances from the nitro groups are 0.2–0.3 Å longer than those from the water molecules and the phenolate groups. The coordinated  $\text{NO}_2$  groups are almost coplanar with the benzene rings. The dihedral angles are in the range 6.72 (18)–26.65 (11)° for the coordinated  $\text{NO}_2$  groups and 40.3 (2)–67 (1)° for the uncoordinated  $\text{NO}_2$  groups.

#### Comment

The investigation of the structural features of the title compound, (I), is part of a series of studies on lanthanide compounds with the 2,6-dinitrophenol (2,6-DNP) ligand. This is the first example of an X-ray crystal structure for a complex of neodymium with the 2,6-DNP ligand.

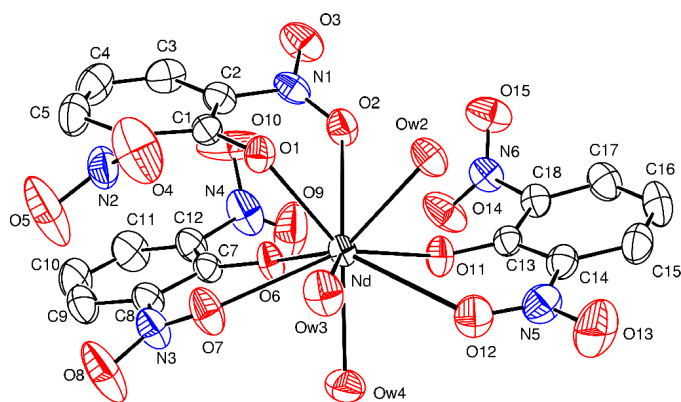


The geometrical features of (I) (Table 1) are similar to our previously reported Y complex (Suh *et al.*, 2002). The  $\text{Nd}^{\text{III}}$  ion is coordinated by nine O atoms (Fig. 1). Three of these neighbours are the O atoms of water molecules (OW2, OW3 and OW4). The  $\text{Nd}-\text{O}_{\text{water}}$  bond distances are in the range 2.433 (3)–2.493 (3) Å. The  $\text{Nd}^{\text{III}}$  ion forms a six-membered chelate ring with each 2,6-DNP ligand, through an O atom of one nitro group (O2, O7 and O12) and the phenolate group (O1, O6 and O11). The  $\text{Nd}-\text{O}$  distances to the nitro groups are in the range 2.661 (3)–2.730 (3) Å, and are longer than those to the phenolates, 2.311 (3)–2.372 (3) Å. The dihedral angles between each benzene ring and its nitro groups are in the range 6.72 (18)–26.65 (11)° for the coordinated nitro groups and 40.3 (2)–67 (1)° for the uncoordinated nitro groups. The closest approach to Nd of the O atoms of the uncoordinated nitro groups is more than 4.5 Å. The coordi-

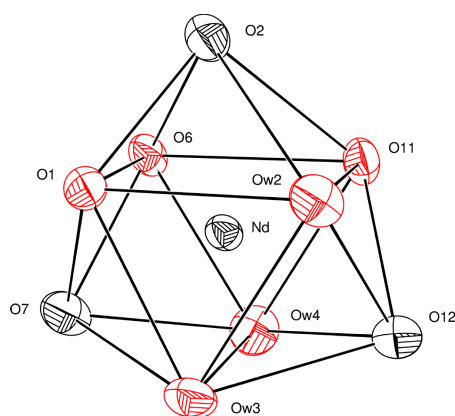
Received 4 April 2002

Accepted 7 May 2002

Online 17 May 2002



**Figure 1**  
ORTEP-3 (Farrugia, 1997) drawing of (I) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 40% probability level. The H atoms of the benzene ring and the water molecules, and second disorder components, have been omitted for clarity.



**Figure 2**  
A view of the tricapped trigonal prismatic geometry around Nd. Atoms are shown as 40% probability ellipsoids.

nation environment of the Nd<sup>III</sup> ion is a slightly distorted tricapped trigonal prism (Fig. 2). Three water molecules and three O atoms (O1, O6 and O11) of 2,6-DNP form a trigonal prism geometry. Another three O atoms (O2, O7, and O12) from the nitro groups cap the sides of the trigonal prism at relatively long distances. In the title compound, the water molecules and the 2,6-DNP ligands are linked by hydrogen bonds into a three-dimensional network (Table 2). For example, the OW2 molecule is connected to the symmetrically related atoms O1, O3 and O4 of 2,6-DNP through hydrogen bonding.

## Experimental

Complex (I) was prepared using a method similar to that of Suh *et al.* (2002). Neodymium chloride solution was prepared by dissolving NdCl<sub>3</sub>·6H<sub>2</sub>O (0.30 g, 0.08 mmol; Aldrich) in distilled water (20 ml) at 333 K with stirring. The pH of the solution was adjusted to about 2 with 7 M HNO<sub>3</sub> solution. The ligand solution was prepared by dissolving 2,6-dinitrophenol (0.3 g, 1.3 mmol; Aldrich) in distilled water (30 ml) at 333 K with stirring. The pH of the ligand solution was adjusted to about 6 with 3 M LiOH solution. The neodymium

chloride solution was slowly added dropwise to the ligand solution. The reaction mixture was stirred for 3 h at 333 K and then cooled to room temperature. Light-yellow crystals of (I) were obtained at room temperature over a period of a few weeks. The complex was recrystallized from distilled water.

## Crystal data

[Nd(C<sub>6</sub>H<sub>3</sub>N<sub>2</sub>O<sub>5</sub>)<sub>3</sub>(H<sub>2</sub>O)<sub>3</sub>]  
*M<sub>r</sub>* = 747.6  
 Triclinic, *P* $\bar{1}$   
*a* = 8.6451 (5) Å  
*b* = 11.8493 (7) Å  
*c* = 13.9227 (11) Å  
 $\alpha$  = 73.800 (5)°  
 $\beta$  = 74.710 (5)°  
 $\gamma$  = 69.236 (5)°  
*V* = 1258.88 (14) Å<sup>3</sup>

*Z* = 2  
*D<sub>x</sub>* = 1.972 Mg m<sup>-3</sup>  
 Mo *K*α radiation  
 Cell parameters from 51 reflections  
 $\theta$  = 4.7–12.6°  
 $\mu$  = 2.16 mm<sup>-1</sup>  
*T* = 293 (2) K  
 Rectangular block, yellow  
 0.40 × 0.30 × 0.20 mm

## Data collection

Bruker *P4* diffractometer  
 2 $\theta$ / $\omega$  scans  
 Absorption correction:  $\psi$  scan  
 (North *et al.*, 1968)  
*T*<sub>min</sub> = 0.469, *T*<sub>max</sub> = 0.649  
 6904 measured reflections  
 5757 independent reflections  
 4661 reflections with *I* > 2σ(*I*)

*R*<sub>int</sub> = 0.026  
 $\theta$ <sub>max</sub> = 27.5°  
*h* = -1 → 11  
*k* = -14 → 15  
*l* = -17 → 18  
 3 standard reflections  
 every 97 reflections  
 intensity decay: none

## Refinement

Refinement on *F*<sup>2</sup>  
*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.034  
*wR* (*F*<sup>2</sup>) = 0.077  
*S* = 1.06  
 5757 reflections  
 424 parameters  
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0273P)^2 + 0.4061P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 1.61 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.78 \text{ e \AA}^{-3}$

**Table 1**

Selected interatomic distances (Å).

Nd—O11	2.311 (3)	N1—O2	1.245 (4)
Nd—O6	2.341 (3)	N2—O4	1.203 (5)
Nd—O1	2.372 (3)	N2—O5	1.205 (5)
Nd—OW3	2.433 (3)	O6—C7	1.284 (4)
Nd—OW4	2.480 (3)	N3—O8	1.226 (5)
Nd—OW2	2.493 (3)	N3—O7	1.240 (5)
Nd—O7	2.661 (3)	O11—C13	1.287 (5)
Nd—O2	2.667 (3)	N5—O12	1.232 (5)
Nd—O12	2.730 (3)	N5—O13	1.236 (5)
O1—C1	1.287 (5)	N6—O14	1.211 (5)
N1—O3	1.226 (4)	N6—O15	1.222 (5)

**Table 2**

Hydrogen-bonding geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
OW2—H18...O3 <sup>i</sup>	0.82 (8)	2.19 (8)	2.939 (5)	152 (7)
OW2—H19...O1 <sup>ii</sup>	0.93 (8)	2.00 (8)	2.889 (4)	161 (6)
OW2—H19...O4 <sup>ii</sup>	0.93 (8)	2.49 (7)	2.976 (5)	113 (5)
OW3—H20...O10 <sup>iii</sup>	0.84 (6)	2.39 (7)	3.17 (3)	155 (5)
OW3—H20...O9 <sup>iii</sup>	0.84 (6)	2.51 (6)	3.28 (2)	154 (5)
OW3—H21...O15 <sup>iii</sup>	0.81 (7)	2.17 (7)	2.862 (5)	144 (7)
OW3—H21...O14 <sup>iii</sup>	0.81 (7)	2.33 (7)	3.088 (5)	156 (7)
OW3—H21...N6 <sup>iii</sup>	0.81 (7)	2.57 (7)	3.365 (5)	166 (7)
OW4—H22...O6 <sup>iv</sup>	0.87	2.12	2.979 (5)	179
OW4—H23...O14 <sup>iv</sup>	0.94	2.45	2.908 (5)	111
OW4—H23...O11 <sup>iv</sup>	0.94	2.48	3.084 (4)	122

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

The positional parameters of the H atoms on the 2,6-DNP rings were calculated geometrically and constrained to ride on their attached atoms, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ . The water H atoms were located from difference Fourier maps and refined freely, except those of OW4, which were fixed. The O9 and O10 atoms in a nitro group are disordered over two positions and the two split atoms are designated by having the suffix *A* after the atom number. The final occupancy factors for the disordered atoms are O9 0.47 (3), O9A 0.53 (3), O10 0.43 (3) and O10A 0.57 (3). The maximum and minimum residuals in the final difference Fourier map were 1.23 Å from O12 and 0.89 Å from Nd, respectively.

Data collection: *XSCANS* (Siemens, 1996); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Bruker, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular

graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

This work is supported financially by the Korean Science and Engineering Foundation (KOSEF R01-2001-00055).

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