

## Triaquatris(2,6-dinitrophenolato- $\kappa^2\text{O}^1, \text{O}^2$ )yttrium(III)

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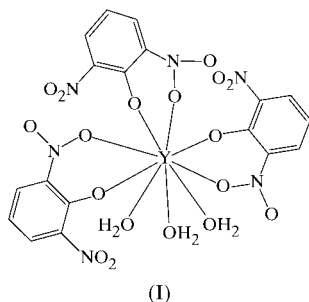
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In the title compound,  $[\text{Y}(\text{C}_6\text{H}_3\text{N}_2\text{O}_5)_3(\text{H}_2\text{O})_3]$ , the Y atom is nine-coordinate with a slightly distorted tricapped trigonal prismatic coordination geometry. The  $\text{Y}^{\text{III}}$  ion is coordinated to three bidentate 2,6-dinitrophenolate ligands and three water molecules. The  $\text{Y}-\text{O}$  bond distances are in the range 2.217 (3)–2.754 (4) Å, with the  $\text{Y}-\text{O}$  distances from the nitro groups being longer than those from the water molecules and the phenol groups. The coordinated  $\text{NO}_2$  groups are almost coplanar with the benzene rings.

### Comment

Very few crystal structures have been reported for transition metal complexes with 2,6-dinitrophenol (2,6-DNP). The known 2,6-DNP complexes with copper show a bimetallic structure bridged by O atoms in sixfold coordination (Zanin *et al.*, 1992). A tricapped trigonal prismatic array around the  $\text{Y}^{\text{III}}$  ion is known for various ligands, such as nonadentate 1,4,7-triazacyclononane (Tei *et al.*, 2000), a macrocyclic ligand and water molecules (Inoue *et al.*, 1994), and just water molecules (Broach *et al.*, 1979). The coordinated water molecules are involved in hydrogen bonds with other ligands. The title complex, (I), is the first example of an X-ray crystal structure for yttrium with the 2,6-DNP ligand.



(I)

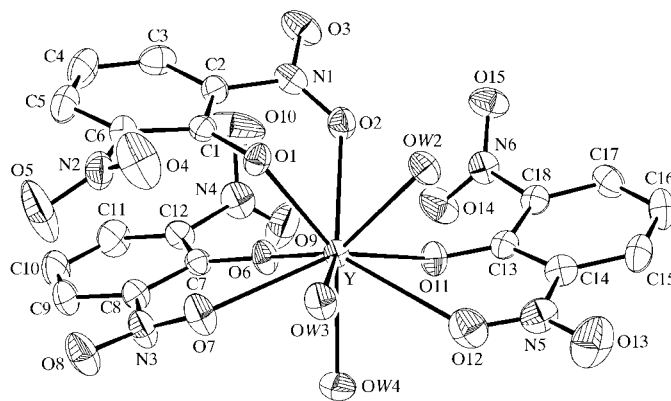
The  $\text{Y}^{\text{III}}$  ion in (I) is coordinated to nine O atoms (Fig. 1). Three of these are the O atoms of water molecules (OW2, OW3 and OW4). The  $\text{Y}-\text{O}_{\text{water}}$  bond distances are within the

range 2.316 (3)–2.389 (3) Å and are shorter than the sum of the covalent radii of Y and O atoms (2.44 Å).

Each 2,6-DNP ligand forms a chelate ring with  $\text{Y}^{\text{III}}$ , through an O atom of one nitro group and the phenol group. The  $\text{Y}-\text{O}$  distances to the nitro groups are in the range 2.605 (3)–2.754 (4) Å, and are rather longer than the others. However, the dihedral angles between the benzene rings and the nitro groups are good evidence for the interaction between the O atoms on the nitro groups and Y.

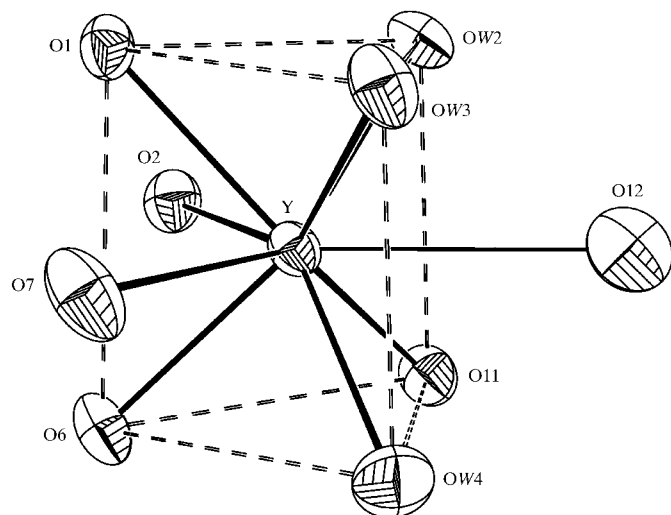
The dihedral angles are in the range 5.92 (18)–15.1 (2)° for the coordinated nitro groups and 49.12 (18)–75 (1)° for the uncoordinated nitro groups. The closest approach to Y of the O atoms of the uncoordinated nitro groups is longer than 4 Å.

There are hydrogen bonds between three water molecules and the O atoms of the 2,6-DNP ligands (Table 2). For example, molecule OW2 is linked to the symmetrically related atoms O1 and O3 through hydrogen bonding.



**Figure 1**

A view of the structure of (I) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 40% probability level and H atoms have been omitted for clarity. Some O atoms in the nitro groups are disordered; only one component is shown.



**Figure 2**

The coordination sphere of the Y atom in (I). The tricapped trigonal prism is indicated by dashed lines.

The skeletal structure around the Y atom forms a slightly distorted tricapped trigonal prism (Fig. 2). Atoms O1, O6 and O11, and the three water molecules form a trigonal prism geometry (shown by dashed lines). At relatively long distances, three O atoms from the nitro groups cap the sides of the trigonal prism.

## Experimental

Complex (I) was synthesized using a method similar to that of Tian *et al.* (1986). Yttrium trichloride solution was prepared by dissolving  $\text{YCl}_3 \cdot 6\text{H}_2\text{O}$  (0.30 g, 0.99 mmol; Aldrich) in distilled water (20 ml) at 333 K with stirring. The pH of the metal solution was adjusted to about 2 with 7 M  $\text{HNO}_3$  solution. The ligand solution was prepared by dissolving 2,6-dinitrophenol (0.30 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 yttrium metal solution was added dropwise slowly to the ligand solution. The reaction mixture was stirred for 3 h at 333 K and then cooled to room temperature. 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

$[\text{Y}(\text{C}_6\text{H}_3\text{N}_2\text{O}_5)_3(\text{H}_2\text{O})_3]$   
 $M_r = 692.27$   
 Triclinic,  $P\bar{1}$   
 $a = 8.5876$  (6) Å  
 $b = 11.7161$  (9) Å  
 $c = 13.8624$  (11) Å  
 $\alpha = 73.531$  (7)°  
 $\beta = 74.745$  (6)°  
 $\gamma = 69.590$  (6)°  
 $V = 1232.44$  (16) Å<sup>3</sup>

$Z = 2$   
 $D_x = 1.865$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 Cell parameters from 53 reflections  
 $\theta = 4.9\text{--}12.5^\circ$   
 $\mu = 2.47$  mm<sup>-1</sup>  
 $T = 301$  (2) K  
 Cubic, yellow  
 $0.42 \times 0.36 \times 0.22$  mm

### Data collection

Bruker P4 diffractometer  
 $2\theta/\omega$  scans  
 Absorption correction:  $\psi$  scan  
 (North *et al.*, 1968)  
 $T_{\min} = 0.385$ ,  $T_{\max} = 0.581$   
 6755 measured reflections  
 5632 independent reflections  
 3771 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.036$   
 $\theta_{\text{max}} = 27.5^\circ$   
 $h = -1 \rightarrow 11$   
 $k = -14 \rightarrow 14$   
 $l = -17 \rightarrow 18$   
 3 standard reflections  
 every 97 reflections  
 intensity decay: none

### Refinement

Refinement on  $F^2$   
 $R(F) = 0.056$   
 $wR(F^2) = 0.103$   
 $S = 1.00$   
 5632 reflections  
 432 parameters

H atoms treated by a mixture of independent and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.0365P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.38$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.42$  e Å<sup>-3</sup>

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 density maps and refined freely. Atoms O9 and O10 of one nitro group were disordered over two positions, and the two split atoms are designated by the suffix *A*. The final occupancy factors for the disordered atoms were O9 0.47 (5) and O9A 0.53 (5), and O10 0.48 (6) and O10A 0.52 (6). The maximum and

**Table 1**

Selected interatomic distances (Å).

Y—O11	2.217 (3)	N1—O2	1.238 (4)
Y—O6	2.246 (3)	N2—O4	1.198 (5)
Y—O1	2.279 (3)	N2—O5	1.205 (4)
Y—OW3	2.316 (3)	O6—C7	1.284 (4)
Y—OW4	2.372 (4)	N3—O8	1.214 (4)
Y—OW2	2.389 (3)	N3—O7	1.239 (4)
Y—O7	2.605 (3)	O11—C13	1.285 (4)
Y—O2	2.608 (3)	N5—O13	1.227 (5)
Y—O12	2.754 (4)	N5—O12	1.244 (5)
O1—C1	1.283 (5)	N6—O14	1.214 (4)
N1—O3	1.221 (4)	N6—O15	1.219 (4)

**Table 2**

Hydrogen-bonding geometry (Å, °).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
OW2—H19 $\cdots$ O1 <sup>i</sup>	0.84 (7)	2.10 (7)	2.920 (4)	164 (6)
OW2—H18 $\cdots$ O3 <sup>ii</sup>	0.81 (7)	2.22 (7)	2.946 (5)	150 (6)
OW3—H20 $\cdots$ O10 <sup>iii</sup>	0.83 (5)	2.21 (6)	3.03 (4)	169 (5)
OW3—H21 $\cdots$ O14 <sup>iii</sup>	0.84 (6)	2.51 (6)	3.115 (5)	130 (5)
OW3—H21 $\cdots$ O15 <sup>iii</sup>	0.84 (6)	1.98 (6)	2.813 (5)	174 (6)
OW4—H22 $\cdots$ O6 <sup>iv</sup>	0.72 (9)	2.40 (9)	3.022 (6)	145 (10)
OW4—H22 $\cdots$ O9 <sup>iv</sup>	0.72 (9)	2.25 (10)	2.794 (17)	133 (10)
OW4—H23 $\cdots$ O14 <sup>iv</sup>	0.69 (8)	2.31 (8)	2.904 (5)	146 (9)

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

minimum residuals in the final difference map were 1.67 Å from C1 and 1.13 Å from N5, 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).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: TA1359). Services for accessing these data are described at the back of the journal.

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