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Diaqua(2,2′-bipyridyl)tris(nitrato-κ^2O,O')-praseodymium(III) 2,2′-bipyridyl sesquisolvate

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Key indicators

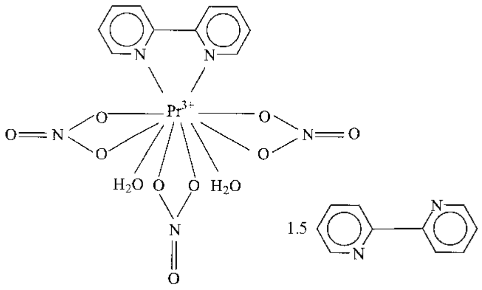
Single-crystal X-ray study
T = 130 K
Mean σ(C–C) = 0.006 Å
R factor = 0.038
wR factor = 0.084
Data-to-parameter ratio = 15.6

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

The Pr atom in the title complex, $[\mathrm{Pr(NO_3)_3(C_{10}H_8N_8)(H_2O)_2}]\cdot1.5\mathrm{C_{10}H_8N_2}$, is ten-coordinate and the relative arrangement of the ligands in the first coordination sphere is affected by the hydrogen bonds between the coordinated water molecules and the uncoordinated 2,2′-bipyridyl (bipy) solvent molecules, one of which lies on a cystallographic inversion centre.

Comment

The Pr atom in the title compound, (I), is ten-coordinate, as shown in Fig. 1. The arrangement in the coordination sphere is similar to that in $[\mathrm{La(NO_3)_3(bipy)(H_2O)_2}]\cdot\mathrm{C_{14}H_{20}O_5}$, (II) (Wen-Xiang Xu*et al.*, 1990), but the ligand orientations are different. The nitrate anions in (I) form dihedral angles of 50.45 (8)°, 69.66 (14)° and 72.61 (9)° with bipy, whereas in (II), the dihedral angles are 30.3°, 37.5° and 46.8°. The difference in the relative orientation of the ligands in (I) and (II) certainly results from the effect of the hydrogen bonds between the coordinated water molecules and the uncoordinated solvent molecules. The occurrence of O—H···N(bipy) and O—H···O(nitrate) hydrogen bonds (Table 2) leads to an intricate two-dimensional hydrogen-bonding network parallel to the *bc* plane (Fig. 2).



(I)

Experimental

The crystals were obtained by slow evaporation at 293 K of 1 ml of a benzene solution containing 25 mg $\mathrm{Pr(NO_3)_3\cdot6H_2O}$ and 4.5 mg 2,2′-bipyridyl ($\mathrm{Pr^{3+}}$:bipy molar ratio of 1:0.5).

Crystal data

$[\mathrm{Pr(NO_3)_3(C_{10}H_8N_8)(H_2O]\cdot}$-
$1.5\mathrm{C_{10}H_8N_2}$
M_r = 753.43
Triclinic, $P\bar{1}$
a = 10.4160 (5) Å
b = 11.4284 (6) Å
c = 14.0585 (10) Å
α = 75.322 (4)°
β = 81.118 (4)°
γ = 64.060 (4)°
V = 1453.96 (15) Å^3
Z = 2
D_x = 1.721 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 13 117 reflections
θ = 2–27.5°
μ = 1.75 mm^{-1}
T = 130 (2) K
Prism, green
0.27 × 0.27 × 0.18 mm

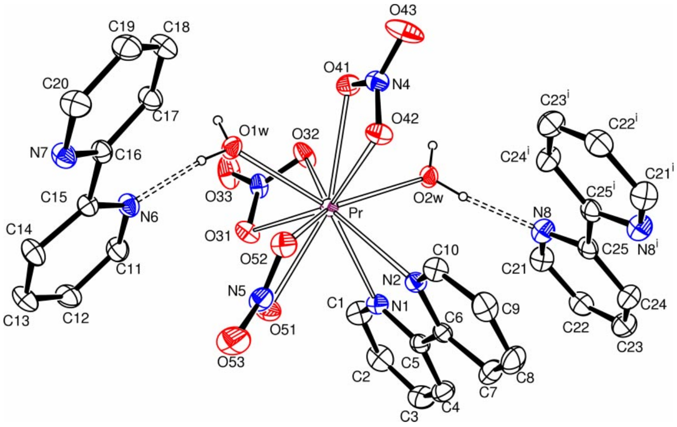


Figure 1
*ORTEP*III (Burnett & Johnson; Farrugia, 1997) view of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme. [Symmetry code: (i) $-x$, $-y$, $-z$.] H atoms have been omitted except on water molecules. Dashed lines indicate hydrogen bonds.

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*MULABS*; Blessing, 1995)
$T_{\min} = 0.659$, $T_{\max} = 0.732$
13117 measured reflections
6527 independent reflections
6042 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.045$
$\theta_{\max} = 27.5°$
$h = -13 \rightarrow 13$
$k = -13 \rightarrow 14$
$l = -16 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.085$
$S = 1.08$
6527 reflections
418 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + 3.9292P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.003$
$\Delta\rho_{\max} = 1.14$ e Å^{-3}
$\Delta\rho_{\min} = -1.89$ e Å^{-3}

Table 1
Selected bond lengths (Å).

Pr—O2*W*	2.461 (2)	Pr—O41	2.618 (2)
Pr—O1*W*	2.468 (2)	Pr—N1	2.668 (3)
Pr—O32	2.544 (3)	Pr—N2	2.676 (3)
Pr—O42	2.554 (2)	Pr···N3	2.997 (3)
Pr—O51	2.568 (2)	Pr···N4	3.014 (3)
Pr—O52	2.582 (3)	Pr···N5	3.000 (3)
Pr—O31	2.601 (3)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H11···O41^{ii}	0.85 (2)	2.00 (3)	2.797 (4)	156 (4)
O1*W*—H12···N6	0.85 (4)	1.88 (2)	2.707 (4)	166 (5)
O2*W*—H21···N8	0.86 (2)	1.90 (2)	2.751 (4)	170 (4)
O2*W*—H22···N7^{iii}	0.84 (3)	2.01 (3)	2.791 (4)	157 (4)

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

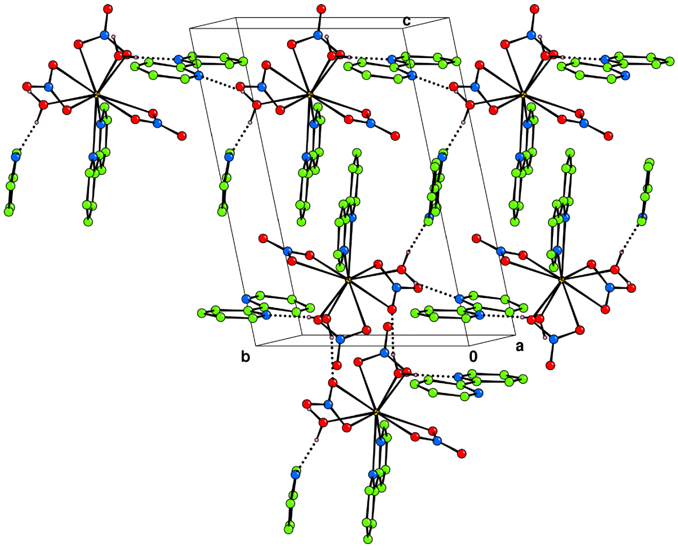


Figure 2
CAMERON (Watkin *et al.*, 1993) view of the packing, showing the O—H···N(bipy) and O—H···O(nitrate) hydrogen-bonding interactions as dashed lines.

All C-bound H atoms were positioned geometrically (C—H = 0.93 Å) and refined using a riding model [$U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C)]. H atoms of water molecules were located in difference Fourier maps and refined with restrained O—H and H···H distances [0.85 (5) and 1.39 (5) Å, respectively] and $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(O). The highest peak and deepest hole are 0.73 and 0.86 Å from the Pr atom, respectively.

Data collection: *KappaCCD Software* (Nonius, 1997); cell refinement: *HKL* (Otwinowski & Minor, 1997); data reduction: *HKL*; program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *ORTEP*III (Burnett & Johnson, 1996); *ORTEP-3 for Windows* (Farrugia, 1997) and *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *SHELXTL* (Sheldrick, 1997).

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