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Bis(2,2′-bipyridyl-*N,N′*)tris(nitrato-*O,O′*)neodymium

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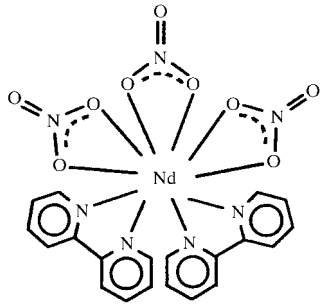
The title compound, [Nd(bipy-*N,N′*)$_2$(NO_3–*O,O′*)$_3$], is found to be isomorphous with the La and Lu analogues having three bidentate nitrate and two bipyridyl ligands giving a ten co-ordinate environment.

Comment

Compounds Ln(bipy)$_2$(NO_3)$_3$ (Ln = La–Lu; bipy = 2,2′-bipyridyl) have been known for many years (Lobanov & Smirnova, 1963; Sinha, 1964; Hart & Laming, 1965). The structures of the La and Lu compounds have more recently been confirmed by X-ray diffraction studies (Al-Kharaghouli & Wood, 1972; Kravchenko, 1972; Kepert *et al.*, 1996). Nd(bipy)$_2$(NO_3)$_3$, (I), is isomorphous with those structures, with three bidentate nitrate and two bipyridyl ligands giving a ten-coordinate environment about the Nd atom. The coordination polyhedron has been described as a bicapped dodecahedron (Al-Kharaghouli & Wood, 1972) or sphenocorona (Kepert *et al.*, 1996). The average Nd—N bond length of 2.596 Å is 0.07 Å shorter than in the La analogue closely corresponding to the 0.05 Å expected on ionic radius grounds (Shannon, 1976). Similarly the average Nd—O bond length of 2.549 Å is 0.05 Å less than that found for the La analogue and is similar to the average value of 2.587 Å found in the 12-coordinate Nd(NO_3)$_3$(18-crown-6) (Bombieri *et al.*, 1980). The O5 atom of the NO_3 group on the twofold axis exhibits very high anisotropic displacement parameters perpendicular to that axis. This may be due to non-planarity of the NO_3 group and consequent disorder of the O5 across the axis which could not be resolved in the presence of the Nd atom.



(I)

It has generally been believed that bipy and phen (1,10-phenanthroline) will not form complexes beyond a 2:1 stoichiometry with Ln(NO_3)$_3$ (Forsberg, 1973; Fréchette, 1992), however, the complexes Ln(bipy)$_3$(NO_3)$_3$ (Ln = Ce, Pr, Nd, Yb) have been reported (Dong *et al.*, 1992) from the reaction of hydrated lanthanide nitrates with 2,2′-bipyridyl (3 moles). A study of the 1:3 Nd:bipy complex was undertaken to clarify this point. Unfortunately all crystals obtained from this reaction were of poor quality with the best data giving R1 = 0.096 (*wR*2 = 0.33, all data) but the structure was Nd(bipy)$_2$(NO_3)$_3$.(bipy). There are no unusual non-bonded contacts between the non co-ordinated bipy molecules and the Nd(bipy)$_2$(NO_3)$_3$ molecules.

Experimental

The title complex (I) was prepared by methods similar to those in the literature (Hart & Laming, 1965). Hot solutions of Nd(NO_3)$_3$·6H_2O (0.219 g, 0.5 mmol) in ethanol (15 ml) and bipy (0.156 g, 1 mmol) in ethanol (15 ml) were mixed. Violet crystals formed overnight.

Crystal data

[Nd(NO_3)$_3$($C_{10}H_8N_2$)$_2$]
M_r = 642.64
Orthorhombic, *Pbcn*
a = 16.935 (3) Å
b = 9.0806 (7) Å
c = 14.987 (3) Å
V = 2304.8 (6) Å^3
Z = 4
D_x = 1.852 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 47 reflections
θ = 5.38–29.99°
μ = 2.32 mm^{-1}
T = 190 (2) K
Block, violet
0.34 × 0.28 × 0.19 mm

Data collection

Siemens P4 diffractometer
ω scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.506, T_{max} = 0.667
3149 measured reflections
2499 independent reflections
1867 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 27.01°
$h = -1 \rightarrow 21$
$k = -1 \rightarrow 11$
$l = -1 \rightarrow 19$
3 standard reflections
every 100 reflections
intensity decay: <1%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0268P)^2 + 3.6161P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.047$	$(\Delta/\sigma)_{max} = 0.010$
$wR(F^2) = 0.129$	$\Delta\rho_{max} = 1.66$ e Å^{-3}
$S = 2.230$	$\Delta\rho_{min} = -2.21$ e Å^{-3}
2499 reflections	
169 parameters	
H-atom parameters constrained	

Data collection: XSCANS (Fait, 1991); cell refinement: XSCANS (Fait, 1991); data reduction: XSCANS (Fait, 1991); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *SHELXL*97 (Sheldrick, 1997).

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