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Tris(3,3'-diamino-2,2'-bipyridine)nickel(II) dinitrate

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Key indicators

Single-crystal X-ray study T = 293 KMean $\sigma(C-C) = 0.008 \text{ Å}$ R factor = 0.061wR factor = 0.099 Data-to-parameter ratio = 13.5

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

In the title complex, $[Ni(C_{10}H_{10}N_4)_3](NO_3)_2$, the cation has crystallographic R32 symmetry while the anion lies on a threefold axis. In the cation, the Ni^{II} atom is coordinated by six N atoms from three 6,6'-diamino-2,2'-bipyridine ligands. In the crystal structure, a two-dimensional network is formed via intermolecular N-H···O hydrogen bonds.

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Comment

2,2'-Bipyridine and its derivatives are very useful ligands from which a great number of complexes have been synthesized. Some of these complexes have been used in dye-sensitized solar cells (Kuang et al., 2006; Ferrere, 2002). We have an interest in complexes containing bipyridine and its derivatives as ligands, and have synthesized a series of complexes with 6,6'-diamino-2,2'-bipyridine as ligand. We report here the structure of the Ni complex, (I) (Fig. 1).

The Ni^{II} atom, located on the intersection of a threefold and three twofold axes, is coordinated in a distorted octahedral ZnN₆ geometry (Table 1). In the 6,6'-diamino-2,2'-bipyridine ligands, which have twofold rotation symmetry, each pyridine ring is essentially planar with a maximum deviation of 0.068 (4) Å for atom C5; the dihedral angle between the two pyridine rings is 32.4 (3)°. This deviation from planarity is expected in terms of steric relief. The anion lies on a threefold axis. In the crystal structure, intermolecular N-H···O hydrogen bonds (Table 2) connect cations and anions, forming a two-dimensional network.

Experimental

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Ni(NO₃)₂·6H₂O (0.0316 g, 0.109 mmol) in H₂O (10 ml) was added to 6,6'-diamino-2,2'-bipyridine (0.0101 g, 0.0542 mmol) in acetonitrile

(5 ml), and the solution was stirred for a few minutes. Yellow crystals of (I) were obtained after allowing the solution to stand at room temperature for one week. The infrared stretching vibrations of the pyridine ring and amino groups appeared at 1638 cm⁻¹, 1465 cm⁻¹ and 1384 cm⁻¹.

Crystal data

$[Ni(C_{10}H_{10}N_4)_3](NO_3)_2$	$D_x = 1.537 \text{ Mg m}^{-3}$
$M_r = 741.39$	Mo $K\alpha$ radiation
Trigonal, R32	$\mu = 0.68 \text{ mm}^{-1}$
a = 14.537 (2) Å	T = 293 (2) K
c = 13.125 (4) Å	Prism, yellow
$V = 2402.2 (9) \text{ Å}^3$	$0.13 \times 0.13 \times 0.05 \text{ mm}$
Z=3	

Data collection

Bruker Smart APEX CCD	4515 measured reflections
diffractometer	1056 independent reflections
φ and ω scans	890 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan	$R_{\rm int} = 0.088$
SADABS (Sheldrick, 1996)	$\theta_{\rm max} = 26.0^{\circ}$
$T_{\text{min}} = 0.917, T_{\text{max}} = 0.967$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0322P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.061$	where $P = (F_0^2 + 2F_c^2)/3$
$wR(F^2) = 0.099$	$(\Delta/\sigma)_{\rm max} < 0.001$
S = 1.12	$\Delta \rho_{\text{max}} = 0.64 \text{ e Å}^{-3}$
1056 reflections	$\Delta \rho_{\min} = -0.35 \text{ e Å}^{-3}$
78 parameters	Absolute structure: Flack (1983),
H-atom parameters constrained	462 Friedel pairs
	Flack parameter: 0.09 (4)

 Table 1

 Selected geometric parameters (\mathring{A} , °).

Ni1-N1	2.081 (3)		
N1 ⁱ -Ni1-N1 ⁱⁱ N1 ⁱ -Ni1-N1	172.7 (3) 78.6 (3)	N1 ⁱⁱ —Ni1—N1 N1 ⁱ —Ni1—N1 ⁱⁱⁱ	96.18 (12) 89.5 (2)
Symmetry codes: (i) $-y+1, x-y+1, z$.	$-x + \frac{2}{3}, -x + y +$	$-\frac{1}{3}$, $-z + \frac{1}{3}$; (ii) $-x + y$,	-x+1, z; (iii)

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

$D-\mathrm{H}\cdots A$	D-H	$H \cdot \cdot \cdot A$	$D \cdot \cdot \cdot A$	$D-\mathrm{H}\cdot\cdot\cdot A$
$N2-H2B\cdots N2^{i}$	0.86	2.37	2.829 (9)	114

Symmetry code: (i) $-x + \frac{2}{3}$, $-x + y + \frac{1}{3}$, $-z + \frac{1}{3}$.

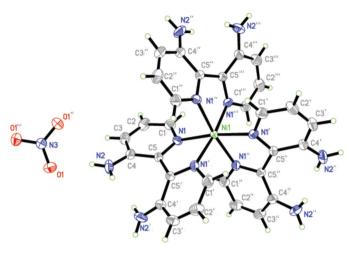


Figure 1

The cation and anion of (I), showing the atom-numbering scheme with displacement ellipsoids drawn at the 30% probability level. [Symmetry codes: (i) $-x + \frac{2}{3}$, $-x + y + \frac{1}{3}$, $-z + \frac{1}{3}$, (ii) -y + 1, x - y + 1, z; (iii) -x + y, -x + 1, z; (iv) $y - \frac{1}{3}$, $x + \frac{1}{3}$, $-z + \frac{1}{3}$; (v) $x - y + \frac{2}{3}$, $-y + \frac{4}{3}$, $-z + \frac{1}{3}$; (vi) -y, x - y, z; (vii) -x + y, -x, z.]

The H atoms were placed in calculated positions and refined in the riding-model approximation, with C—H = 0.93 Å, $U_{\rm iso}({\rm H})$ = 1.2_{eq}(C); N—H = 0.86 Å, $U_{\rm iso}({\rm H})$ = 1.2_{eq}(N).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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